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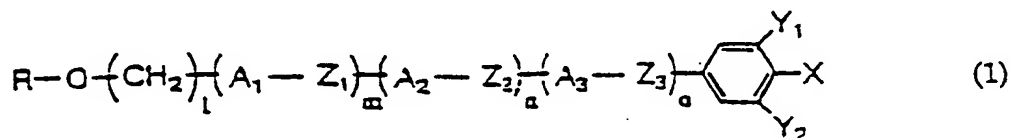
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(54) Ester derivate, liquid crystal composition and liquid crystal display element

(57) A novel liquid crystalline compound having a large dielectric anisotropy value, a small change in the threshold voltage depending upon temperatures, and an improved solubility in other liquid crystal materials at low temperatures, and a liquid crystal composition containing the compound are provided,
which liquid crystalline compound is expressed by the formula (1)



wherein R is H or an alkyl group of 1 to 10C; ℓ is an integer of 1 to 9; m, n and o are each independently 0 or 1; A₁, A₂ and A₃ are each independently trans-1,4-cyclohexylene group, 1,4-phenylene group, one or more H atoms on the ring of which group may be replaced by F, or the like group; Z₁, Z₂ and Z₃ are each independently -COO-, etc., but at least one member of them is -COO-; X is CF₃, etc.; and Y₁ and Y₂ are each independently H or F.

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Description

BACKGROUND OF THE INVENTION

1. Field of the Invention

This invention relates to a liquid crystalline compound and a liquid crystal composition, and more particularly, it relates to a novel ester compound having a 4-substituted phenyl group, a 3-fluoro-4-substituted phenyl group or a 3,5-difluoro-4-substituted phenyl group, a liquid crystal composition containing the compound, and a liquid crystal composition composed by using the liquid crystal composition.

Description of the Related Art

Display elements using liquid crystalline compounds have been broadly utilized for watch, electric calculator, word processor, etc. These display elements are those utilizing the optical anisotropy, dielectric anisotropy, etc. of liquid crystalline compounds.

Liquid crystal phases include nematic liquid crystal phase, smectic liquid crystal phase and cholesteric liquid crystal phase, and among these, materials utilizing nematic liquid crystal phase have been most broadly used. Further, display modes include dynamic scattering (DS) mode, deformation of vertical aligned phase (DAP) mode, guest-host (GH) mode, twisted nematic (TN) mode, super-twisted nematic (STN) mode, thin-film transistor (TFT) mode, etc.

Liquid crystalline compounds used in these display modes are necessary to exhibit liquid crystal phases within a broad temperature range around room temperature, has a sufficient stability under conditions under which display elements are used; and have characteristics sufficient for driving display elements, but at present, a single liquid crystalline compound satisfying these conditions has not yet been found. Thus, it is an actual state that liquid crystal compositions provided with required characteristics have been prepared by blending liquid crystalline compounds of several kinds to several tens kinds and if necessary, further non-liquid crystalline compounds. It is required for these liquid crystal compositions to be stable to light, heat and air which are usually present under conditions wherein display elements are used, and further to be stable to electric field or electromagnetic irradiation, and still further to be chemically stable to compounds to be blended. Further, it is required for liquid crystal compositions that the values of various physical properties such as optical anisotropy value (Δn), dielectric anisotropy value ($\Delta \epsilon$), etc. are suitable, depending upon the display mode and the shape of the display element. Further, the respective components in the liquid crystal compositions are important to have a good compatibility with each other.

Among these requirements, there has been increasing a requirement for further reduction in the threshold voltage greatly contributing to a high speed response necessary for making a large picture surface of liquid crystal display elements. A liquid crystalline compound having a large $\Delta \epsilon$ value is necessary therefor. (E. Jakeman et al, Phys. Lett., 39A. 69 (1972)).

Further, in order to make a high picture surface quality possible, a liquid crystalline compound having a small change in the threshold voltage depending upon temperatures is needed.

In order to achieve these objects, compounds of the formulas (a) and (b) are respectively disclosed in Japanese patent application Hyo No. Hei 3-503637 and Japanese patent application laid-open No. Hei 4-279560. Further, compounds disclosed in Japanese patent application laid-open No. Sho 55-40660, Japanese patent application Hyo No. Hei 2-501311, and Japanese patent application Hyo No. Hei 3-500413 are known.

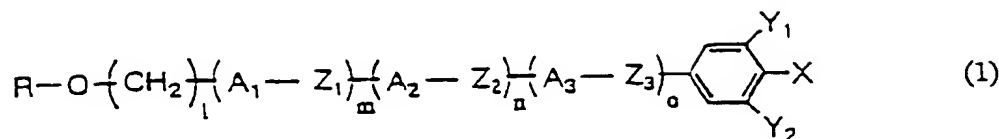
However, these compounds have problems that change in the threshold voltage depending upon temperatures can not be yet regarded as being sufficiently small and further the solubility thereof in liquid crystal compositions at low temperatures is insufficient.

SUMMARY OF THE INVENTION

The object of the present invention is to provide an ester compound which has overcome the above drawbacks of the prior art, and has a particularly large $\Delta \epsilon$, a small change in the driving voltage depending upon temperatures, and an improved solubility in other liquid crystal compositions at low temperatures; a liquid crystal composition containing the above compound; and a liquid crystal display element composed using the liquid crystal composition.

The present invention has the following aspects of items (1) to (13):

(1) A liquid crystalline compound expressed by the formula (1):



wherein R represents an alkyl group of 1 to 10 carbon atoms; ℓ represents an integer of 1 to 9; m, n and o each independently represent 0 or 1; A_1 , A_2 and A_3 each independently represent trans-1,4-cyclohexylene group, or 1,4-phenylene group, pyrimidine-2,5-diyl group, pyridine-2,5-diyl group or 1,3-dioxane-2,5-diyl group, one or more hydrogen atoms on the rings of which groups may be replaced by F; Z_1 , Z_2 and Z_3 each independently represent -COO-, $-(\text{CH}_2)_2$ - or a covalent bond at least one of which represent -COO-; X represents CN, CF_3 , CF_2H , CFH_2 , OCF_3 or OCF_2H ; Y_1 and Y_2 each independently represent H or F, but when X represents CN, Y_1 and Y_2 both represent F.

(2) A liquid crystalline compound according to item (1), wherein m represents 1; n and o represents 0; A_1 represents 1,4-phenylene group, one or more hydrogen atoms on the ring of which group may be replaced by F; and Z_1 represents -COO-.

(3) A liquid crystalline compound according to item (1), wherein m and n each represent 1; o represents 0; A_1 and A_2 each represent 1,4-phenylene group, one or more hydrogen atoms on the ring of which group may be replaced by F; and Z_1 represents -COO-.

(4) A liquid crystalline compound according to item (1), wherein m and n each represent 1; o represents 0; A_1 represents 1,4-phenylene group, one or more hydrogen atoms on the ring of which group may be replaced by F; A_2 represents trans-1,4-cyclohexylene group; and Z_1 represents -COO-.

(5) A liquid crystalline compound according to item (1), wherein m and n each represent 1; o represents 0; A_1 represents trans-1,4-cyclohexylene group; A_2 represents 1,4-phenylene group, one or more hydrogen atoms on the ring of which group may be replaced by F; and Z_2 represents -COO-.

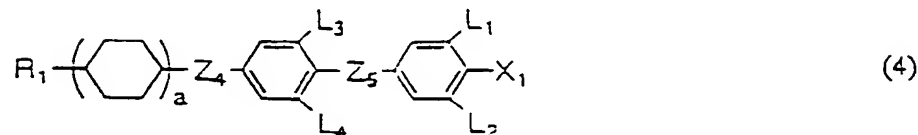
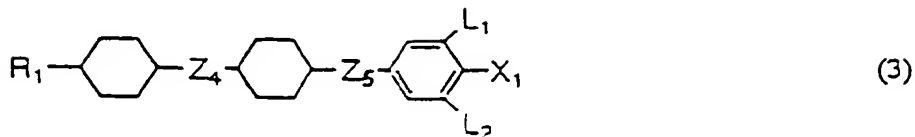
(6) A liquid crystalline compound according to item (1), wherein m and n each represent 1; o represents 0; A_1 and A_2 each represent 1,4-phenylene group, one or more hydrogen atoms on the ring of which group may be replaced by F; and Z_2 represents -COO-.

(7) A liquid crystalline compound according to item (1), wherein m and n each represent 1; o represents 0; A_1 represent trans-1,4-cyclohexylene; A_2 represents 1,4-phenylene group, one or more hydrogen atoms on the ring of which group may be replaced by F; Z_1 represents $-(\text{CH}_2)_2$ -; and Z_2 represents -COO-.

(8) A liquid crystalline compound according to item (1), wherein m, n and o each represent 1; A_1 represents trans-1,4-cyclohexylene group; A_2 and A_3 each represent 1,4-phenylene group, one or more hydrogen atoms of on the ring of which group may be replaced by F; Z_1 and Z_3 each represent a covalent bond; and Z_2 represents -COO-.

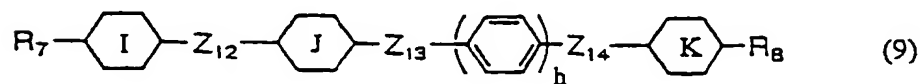
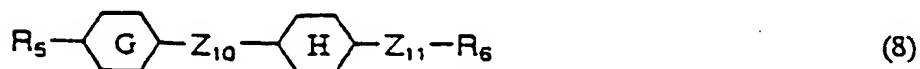
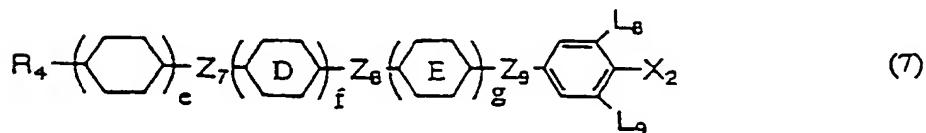
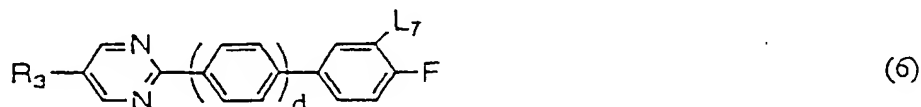
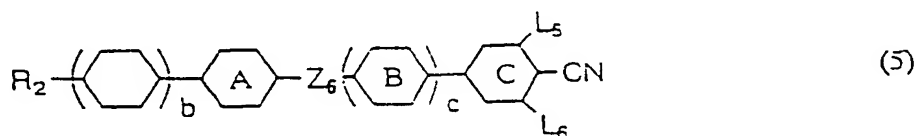
(9) A liquid crystal composition characterized by containing at least one member of the liquid crystalline compound set forth in either one of items (1) to (8).

(10) A liquid crystal composition characterized by containing as a first component, at least one member of the liquid crystalline compounds set forth in either one of items (1) to (8), and as a second component, at least one member selected from the group consisting of compounds expressed by the following formulas (2), (3) and (4):



In these formulas, R_1 represents an alkyl group of 1 to 10 carbon atoms; X_1 represents F, Cl, OCF_3 , OCF_2H , CF_3 , CF_2H or CFH_2 ; L_1 , L_2 , L_3 and L_4 each independently represent H or F; Z_4 and Z_5 each independently represent $-(\text{CH}_2)_2-$, $-\text{CH}=\text{CH}-$ or a covalent bond; and a represents 1 or 2.

(11) A liquid crystal composition characterized by containing as a first component, at least one member of the liquid crystalline compounds set forth in either one of items (1) to (8), and as a second component, at least one member selected from the group consisting of compounds expressed by the following formulas (5), (6), (7), (8) and (9):



In the formula (5), R_2 represents F, an alkyl group of 1 to 10 carbon atoms or an alkenyl group of 2 to 10 carbon atoms, and one or more non-adjacent methylene groups $(-\text{CH}_2-)$ in the alkyl group or the alkenyl group may be

replaced by oxygen (-O-); ring A represents trans-1,4-cyclohexylene group, 1,4-phenylene group, pyrimidine-2,5-diyl group, or 1,3-dioxane-2,5-diyl group; ring B represents trans-1,4-cyclohexylene group, 1,4-phenylene group or pyrimidine-2,5-diyl group; ring C represents trans-1,4-cyclohexylene group or 1,4-phenylene group; Z₆ represents -(CH₂)₂-, -COO- or a covalent bond; L₅ and L₆ each independently represent H or F; b and c each independently represent 0 or 1.

In the formula (6), R₃ represents an alkyl group of 1 to 10 carbon atoms; L₇ represents H or F; and d represents 0 or 1.

In the formula (7), R₄ represents an alkyl group of 1 to 10 carbon atoms; rings D and E each independently represent trans-1,4-cyclohexylene group or 1,4-phenylene group; Z₇ and Z₈ each independently represent -COO- or a covalent bond; Z₉ represents -COO- or -C=C-; L₈ and L₉ each independently represent H or F; X₂ represents F, OCF₃, OCF₂H, CF₃, CF₂H or CFH₂, but when X₂ represents OCF₃, OCF₂H₂, CF₃, CF₂H or CFH₂, L₈ and L₉ both represent H; and e, f and g each independently represent 0 or 1.

In the formula (8), R₅ and R₆ each independently represent an alkyl group of 1 to 10 carbon atoms or an alkenyl group of 2 to 10 carbon atoms, and in either of the groups, one or more non-adjacent methylene groups (-CH₂-) may be replaced by oxygen (-O-);

ring G represents trans-1,4-cyclohexylene group, 1,4-phenylene group or pyrimidine-2,5-diyl group; ring H represents trans-1,4-cyclohexylene group or 1,4-phenylene group; Z₁₀ represents -C=C-, -COO-, -(CH₂)₂-, -CH=CH-C=C- or a covalent bond; and Z₁₁ represents -COO- or a covalent bond.

In the formula (9), R₇ and R₈ each independently represent an alkyl group of 1 to 10 carbon atoms or an alkenyl group of 2 to 10 carbon atoms, and in either of the groups, one or more non-adjacent methylene groups (-CH₂-) may be replaced by oxygen (-O-); ring I represents trans-1,4-cyclohexylene group, 1,4-phenylene group or pyrimidine-2,5-diyl group; ring J represents trans-1,4-cyclohexylene group, or 1,4-phenylene group, or pyrimidine-2,5-diyl group, one or more hydrogen atoms on the rings of which groups may be replaced by F, ring K represents trans-1,4-cyclohexylene group or 1,4-phenylene group; Z₁₂ and Z₁₄ each independently represent -COO-, -(CH₂)₂- or a covalent bond; Z₁₃ represents -CH=CH-, -C=C-, -COO- or a covalent bond; and h represents 0 or 1.

(12) A liquid crystal composition, characterized by containing as a first component, at least one member of the liquid crystalline compounds set forth in either one of items (1) to (8), as a part of a second component, at least one member selected from the group consisting of the compounds expressed by the formulas (2), (3) and (4) set forth in item (10), and as another part of the second component, at least one member selected from the group consisting of the compounds expressed by the formulas (5), (6), (7), (8) and (9) set forth in item (11).

(13) A liquid crystal display element composed of a liquid crystal composition set forth in either one of items (9) to (12).

(14) A phenol derivative as a raw material for a liquid crystalline compound, expressed by the formula (1-a):



wherein A₃ represents trans-1,4-cyclohexylene group or 1,4-phenylene group one or more of hydrogen atoms on the ring of which group may be replaced by F, X represents CF₃, CF₂H, CFH₂, OCF₃ or OCF₂H, Y₁ represents H or F, o represents an integer of 0 or 1, but when o represents 0, and X represents CF₃, Y₁ represents H.

BRIEF DESCRIPTION OF THE ACCOMPANYING DRAWINGS

Fig. 1 refers to a graph for explaining the effectiveness of a liquid crystal composition example of the present invention.

Fig. 2 refers to a graph explaining the effectiveness of another liquid crystal composition of the present invention.

Descriptions of the symbols:

- (a) A liquid crystal composition example of the present invention.
- (b) Another liquid crystal composition example of the present invention.
- (c) A comparative liquid crystal composition example.
- (d) another comparative liquid crystal example.

DESCRIPTION OF PREFERRED EMBODIMENTS

The liquid crystalline compound of the present invention expressed by the formula (1) has a large $\Delta\epsilon$, a small change in the threshold voltage depending upon temperatures and a good solubility in other liquid crystal compositions even at low temperatures. Further, these liquid crystalline compounds are physically and chemically sufficiently stable under conditions where display elements are usually used, and further, when the six-membered ring, substituent and/or bonding group, among the molecule-constituting elements are suitably selected, it is possible to derive them into those having desired physical properties. Thus, when the compound of the present invention is used as a component of liquid crystal composition, it is possible to provide a novel liquid crystal composition having preferred characteristics.

The compound of the present invention expressed by the formula (1) is classified into those expressed by the following formulas (1a) to (1l):

Compound having 2 six-membered rings:



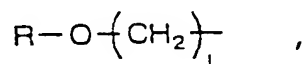
Compounds having 3 six-membered rings:



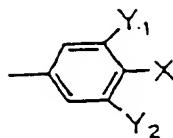
Compounds having 4 six-membered rings:



In these formulas, R_9 is a group expressed below:



Q is a group expressed below:



The compounds expressed by the formula (1a) are further developed into the following formulas (1aa) to (1ae):

R₉-Cyc-COO-Q (1aa)

R₉-Phe-COO-Q (1ab)

R₉-Pyr-COO-Q (1ac)

R₉-Pyd-COO-Q (1ad)

R₉-Dio-COO-Q (1ae)

Among these compounds, those expressed by (1aa) and (ab) are particularly preferred.

In the above formulas, Cyc represents trans-1,4-cyclohexylene group; Phe represents 1,4-phenylene group; Pyr represents pyrimidine-2,5-diyl group; Pyd represents pyridine-2,5-diyl group; Dio represents 1,3-dioxane-2,5-diyl group; one or more hydrogen atoms of the Phe may be replaced by F; A₁, A₂ and A₃ are selected from the group consisting of Cyc, Phe, Pyr, Pyd and Dio, and preferably two or more Pyr, Pyd, Dio are not contained in the molecule.

Compounds expressed by the formula (1b) are developed into those expressed by the following formulas (1ba) to (1bj):

R₉-Cyc-Cyc-COO-Q (1ba)

R₉-Cyc-Phe-COO-Q (1bb)

R₉-Phe-Cyc-COO-Q (1bc)

R₉-Phe-Phe-COO-Q (1bd)

R₉-Pyr-Phe-COO-Q (1be)

R₉-Pyr-Cyc-COO-Q (1bf)

R₉-Pyd-Phe-COO-Q (1bg)

R₉-Pyd-Cyc-COO-Q (1bh)

R₉-Dio-Phe-COO-Q (1bi)

R₉-Dio-Cyc-COO-Q (1bj)

Among these compounds, those expressed by the formulas (1ba) to (1be), (1bg) and (1bi) are particularly preferred.

The compounds expressed by the formula (1c) are further developed into those expressed by the following formulas (1ca) to (1cn):

R₉-Cyc-COO-Cyc-Q (1ca)

R₉-Cyc-COO-Phe-Q (1cb)

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	R ₉ -Cyc-COO-Pyr-Q	(1cc)
	R ₉ -Cyc-COO-Pyd-Q	(1cd)
5	R ₉ -Phe-COO-Cyc-Q	(1ce)
	R ₉ -Phe-COO-Phe-Q	(1cf)
10	R ₉ -Phe-COO-Pyr-Q	(1cg)
	R ₉ -Phe-COO-Pyd-Q	(1ch)
	R ₉ -Pyr-COO-Phe-Q	(1ci)
15	R ₉ -Pyr-COO-Cyc-Q	(1cj)
	R ₉ -Pyd-COO-Phe-Q	(1ck)
	R ₉ -Pyd-COO-Cyc-Q	(1cl)
20	R ₉ -Dio-COO-Phe-Q	(1cm)
	R ₉ -Dio-COO-Cyc-Q	(1cn)

25 Among these compounds, those expressed by the formulas (1ca), (1cb), (1ce) and (1cf) are particularly preferred.
The compounds expressed by the formula (1d) are further developed into those expressed by the following formulas (1da) to (1dk):

30	R ₉ -Cyc-(CH ₂) ₂ -Cyc-COO-Q	(1da)
	R ₉ -Cyc-(CH ₂) ₂ -Phe-COO-Q	(1db)
	R ₉ -Phe-(CH ₂) ₂ -Cyc-COO-Q	(1dc)
35	R ₉ -Phe-(CH ₂) ₂ -Phe-COO-Q	(1dd)
	R ₉ -Phe-(CH ₂) ₂ -Pyr-COO-Q	(1de)
40	R ₉ -Pyr-(CH ₂) ₂ -Phe-COO-Q	(1df)
	R ₉ -Pyr-(CH ₂) ₂ -Cyc-COO-Q	(1dg)
	R ₉ -Pyd-(CH ₂) ₂ -Phe-COO-Q	(1dh)
45	R ₉ -Pyd-(CH ₂) ₂ -Cyc-COO-Q	(1di)
	R ₉ -Dio-(CH ₂) ₂ -Phe-COO-Q	(1dj)
50	R ₉ -Dio-(CH ₂) ₂ -Cyc-COO-Q	(1dk)

Among these compounds, compounds expressed by the formulas (1da) to (1dd) are particularly preferred.
The compounds expressed by the formula (1e) are further developed into compounds expressed by the following formulas (1ea) to (1ek):

55	R ₉ -Cyc-COO-Cyc-COO-Q	(1ea)
	R ₉ -Cyc-COO-Phe-COO-Q	(1eb)
	R ₉ -Phe-COO-Cyc-COO-Q	(1ec)

	R ₉ -Phe-COO-Phe-COO-Q	(1ed)
	R ₉ -Phe-COO-Pyr-COO-Q	(1ee)
5	R ₉ -Pyr-COO-Phe-COO-Q	(1ef)
	R ₉ -Pyr-COO-Cyc-COO-Q	(1eg)
10	R ₉ -Pyr-COO-Phe-COO-Q	(1eh)
	R ₉ -Pyr-COO-Cyc-COO-Q	(1ei)
	R ₉ -Dio-COO-Phe-COO-Q	(1ej)
15	R ₉ -Dio-COO-Cyc-COO-Q	(1ek)

Among these compounds, compounds expressed by the formulas (1ea) to (1ed) are particularly preferred.

The compounds expressed by the formula (1f) are further developed into compounds expressed by the following formulas (1fa) to (1fh):

20	R ₉ -Cyc-COO-Cyc-Cyc-Q	(1fa)
	R ₉ -Cyc-COO-Cyc-Phe-Q	(1fb)
25	R ₉ -Cyc-COO-Phe-Phe-Q	(1fc)
	R ₉ -Cyc-COO-Phe-Cyc-Q	(1fd)
	R ₉ -Phe-COO-Cyc-Cyc-Q	(1fe)
30	R ₉ -Phe-COO-Cyc-Phe-Q	(1ff)
	R ₉ -Phe-COO-Phe-Cyc-Q	(1fg)
35	R ₉ -Phe-COO-Phe-Phe-Q	(1fh)

Among these compounds, compounds expressed by the formulas (1fb), (1fc), (1ff) and (1fh) are particularly preferred.

The compounds expressed by the formula (1g) are further developed into compounds expressed by the following formulas (1ga) to (1gh):

	R ₉ -Cyc-Cyc-COO-Cyc-Q	(1ga)
	R ₉ -Cyc-Cyc-COO-Phe-Q	(1gb)
45	R ₉ -Cyc-Phe-COO-Cyc-Q	(1gc)
	R ₉ -Cyc-Phe-COO-Phe-Q	(1gd)
50	R ₉ -Phe-Cyc-COO-Cyc-Q	(1ge)
	R ₉ -Phe-Cyc-COO-Phe-Q	(1gf)
	R ₉ -Phe-Phe-COO-Cyc-Q	(1gg)
55	R ₉ -Phe-Phe-COO-Phe-Q	(1gh)

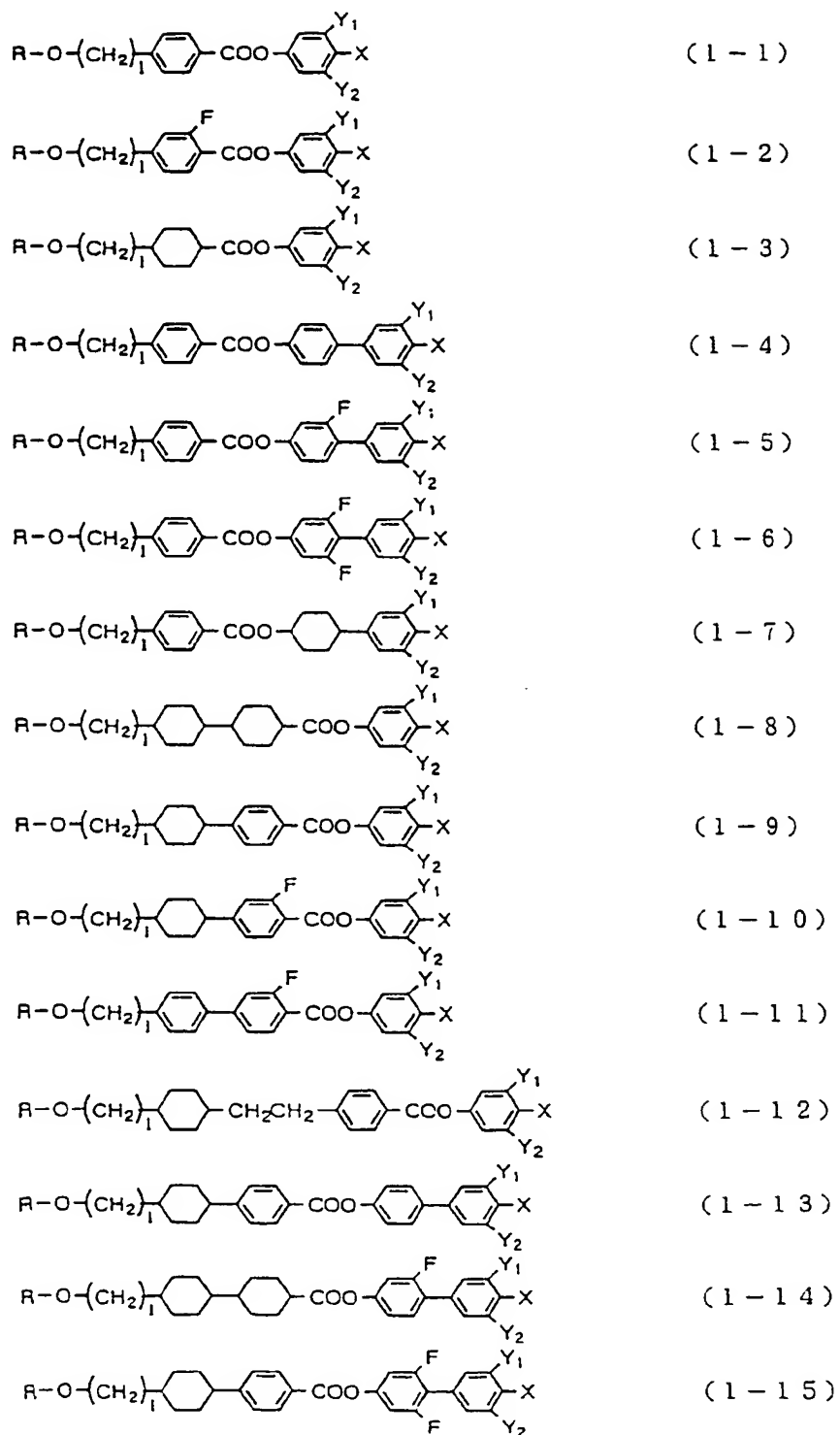
Among these compounds, those expressed by the formulas (1gb), (1gc) and (1gd) are particularly preferred.

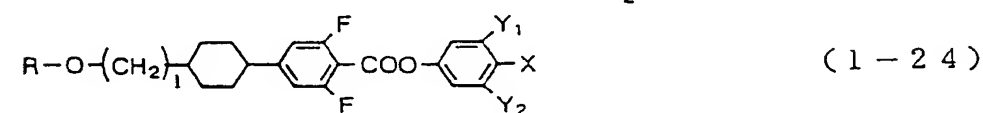
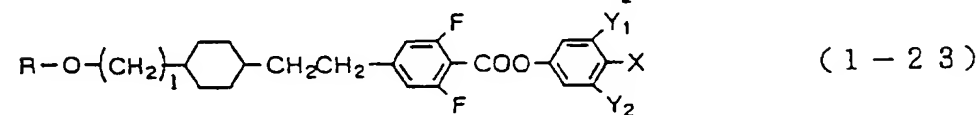
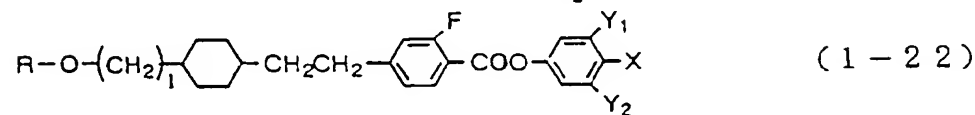
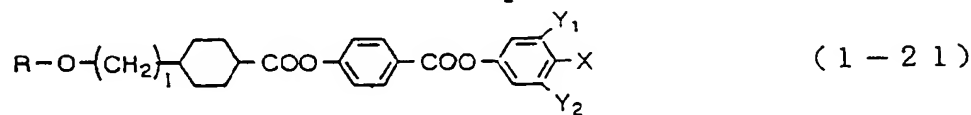
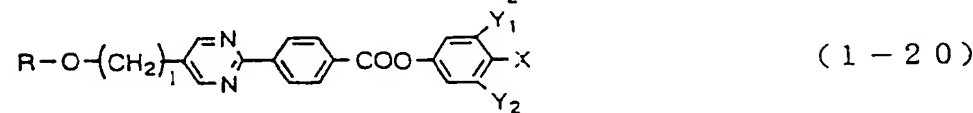
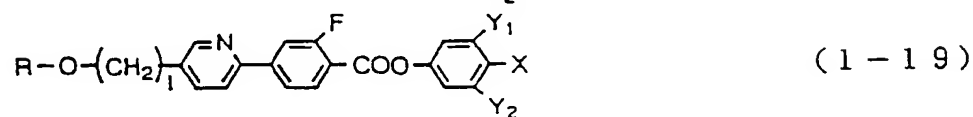
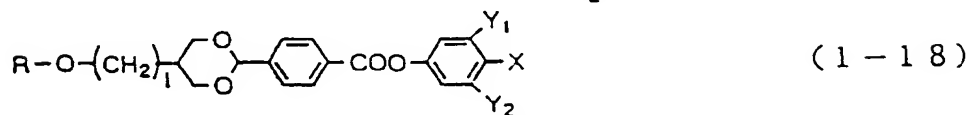
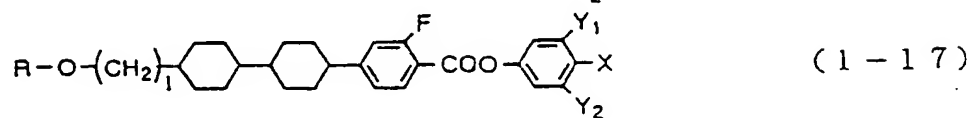
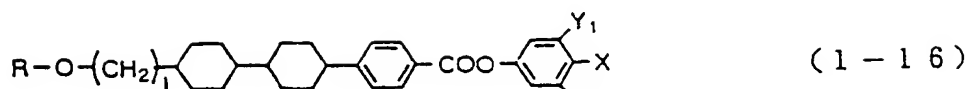
The compounds expressed by the formula (1h) are further developed into compounds expressed by the following formulas (1ha) to (1hh):

5	R_9 -Cyc-Cyc-Cyc-COO-Q	(1ha)
	R_9 -Cyc-Cyc-Phe-COO-Q	(1hb)
	R_9 -Cyc-Phe-Phe-COO-Q	(1hc)
10	R_9 -Cyc-Phe-Cyc-COO-Q	(1hd)
	R_9 -Phe-Cyc-Cyc-COO-Q	(1he)
	R_9 -Phe-Phe-Cyc-COO-Q	(1hf)
15	R_9 -Phe-Cyc-Phe-COO-Q	(1hg)
	R_9 -Phe-Phe-Phe-COO-Q	(1hh)

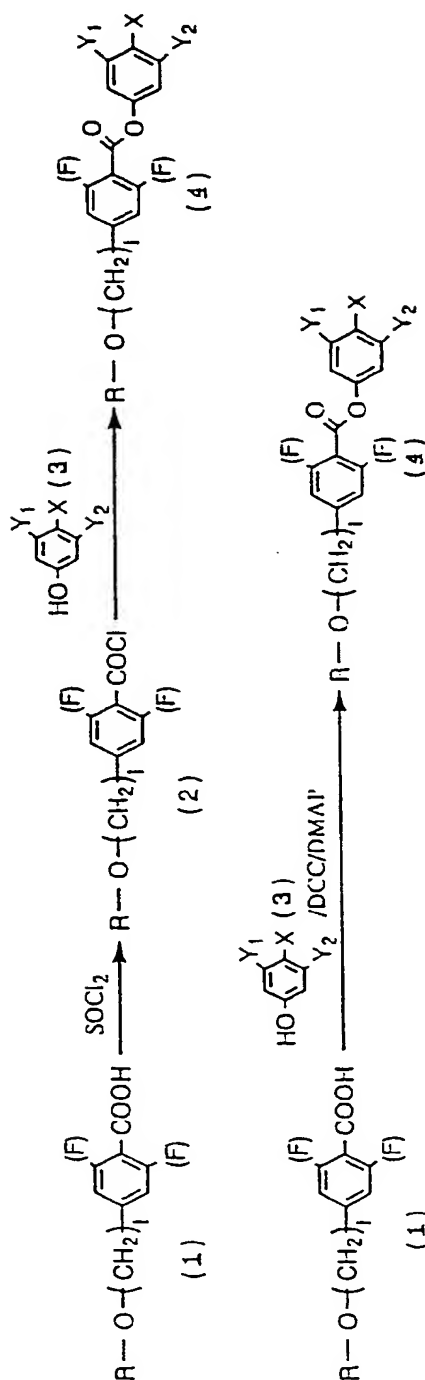
20 In all of the above mentioned compounds, R_9 represents an alkoxyalkyl group of 2 to 19 carbon atoms, and particularly preferred groups among them are methoxymethyl, methoxyethyl, methoxypropyl, methoxybutyl, methoxypentyl, methoxynonyl, ethoxymethyl, ethoxyethyl, ethoxypropyl, ethoxybutyl, propoxymethyl, propoxyethyl, propoxypropyl, butoxymethyl, butoxyethyl, pentyloxymethyl, hexyloxymethyl, heptyloxymethyl and decyloxymethyl.

25 As described above, compounds expressed by the formulas, (1aa), (1ab), (1ba), (1bb), (1bc), (1bd), (1be), (1bg), (1bi), (1ca), (1cb), (1ce), (1cf), (1da), (1db), (1dc), (1dd), (1ea), (1eb), (1ec), (1ed), (1fb), (1fc), (1ff), (1fh), (1gb), (1gc), (1gd) and (1hb) can be said to be particularly preferred, but among them as more preferred compounds, compounds expressed by the following formulas (1-1) to (1-24) can be mentioned:





The liquid crystalline compound of the present invention expressed by the formula (1) can be prepared according to known, general organic synthesis process. For example, it can be conveniently prepared according to the following process:



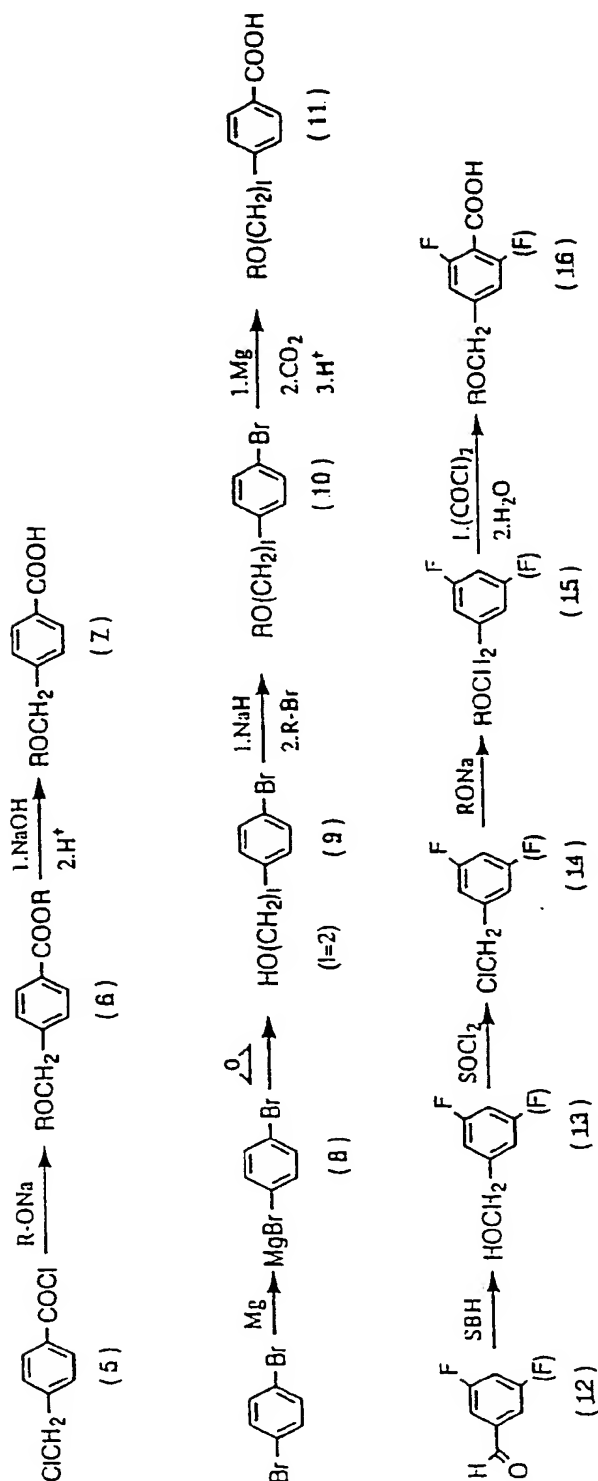
Namely, it is possible to prepare the compound of the formula (1), by esterification such as dehydration condensa-

In more details, a carboxylic acid derivative (1) is converted into an acid halide (2), by means of a halogenating reagent such as thionyl chloride etc., in a solvent such as toluene, benzene, etc. or in the absence of solvent, followed by reacting (2) with a phenol derivative (3) in a solvent such as toluene, benzene, etc., to obtain the objective compound example (4). The reactions in a series is preferably carried out at a temperature between room temperature and the

boiling point of solvent, and further in the presence of a base such as pyridine, triethylamine, dimethylaniline, tetramethyl urea, etc. (E.J. Corey et al, Journal of Organic Chemistry, 38, 3223 (1973), C. Raha, Organic Synthesis, IV, 263 (1963), B. Iselin et al, Helvetica Chimica Acta, 40, 373 (1957), M.S. Newman et al, Tetrahedron Letters, 3267 (1967)).

Alternatively, when a carboxylic acid derivative (1) is reacted with a phenol derivative (3), in a solvent such as dichloromethane, chloroform, etc., and in the presence of dicyclohexylcarbodiimide (hereinafter abbreviated to DCC) and dimethylaminopyrimidine (hereinafter abbreviated to DMAP), it is also possible to obtain the objective compound exmaple (4) (B. Neises et al, Organic Synthesis, 63, 183 (1985)).

The carboxylic acid derivative (1) as one of the raw materials can be prepared for example according to the following process:



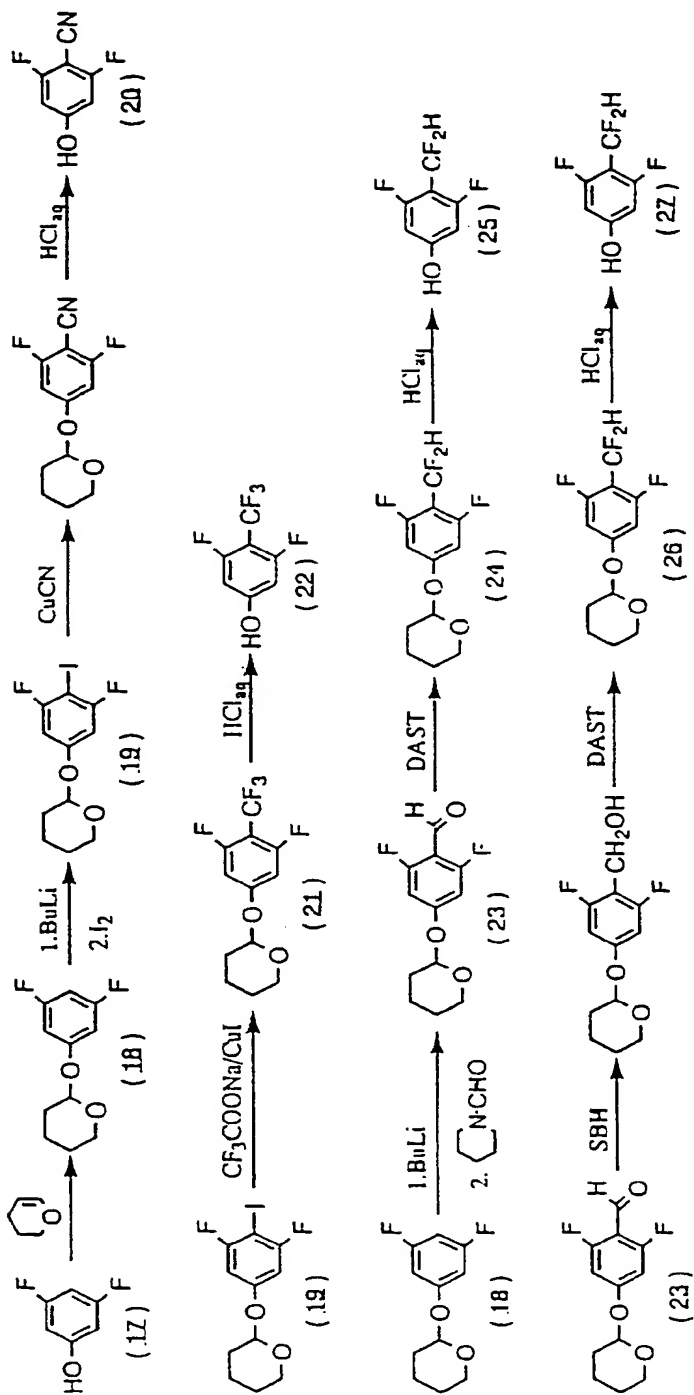
Namely, when compound (5) is reacted with a sodium alkoxide, to obtain a compound (6), followed by hydrolysis in the presence of a base such as NaOH, KOH, etc., it is possible to obtain a carboxylic acid derivative example (Z).

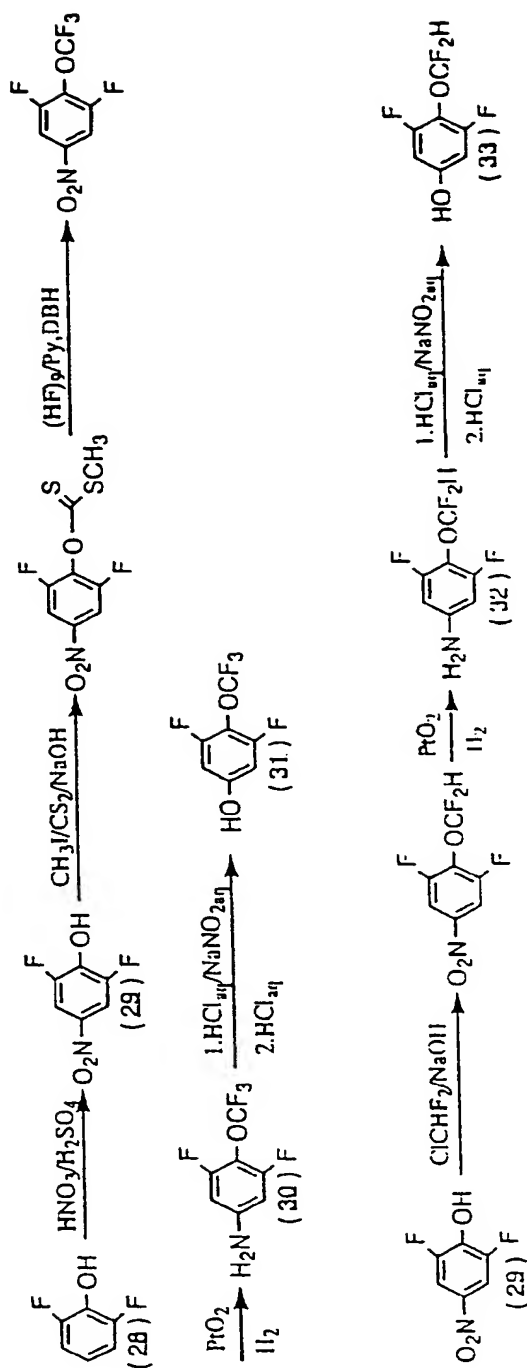
Further, when a Grignard reagent (8), of a dihalogenobenzene is prepared, followed by reacting it with ethylene oxide, to obtain an alcohol (9) and then an ether (10), preparing a Grignard reagent of the ether and reacting it with CO₂, it is possible to obtain a carboxylic acid derivative example (11).

In the case of the alcohol (9), compounds having an ℓ of 3 or more can be obtained by reacting with the corresponding ring-form ether in place of ethylene oxide. Alternatively, (9) can be easily obtained by carrying out carbon number-increasing reaction according to a known, general organic synthesis process, for example, to prepare the corresponding derivative of carboxylic acid, aldehyde, ester, etc., followed by reducing the derivative with a reducing agent such as sodium boron hydride (SBH), borane, lithium aluminum hydride (LAH), sodium bis(2-methoxyethoxy)aluminum hydride (SBMEA, Red-Al) or the like.

Alternatively, it is possible to obtain the carboxylic acid derivative example (16), by reducing aldehyde (12) with a reducing agent such as SBH, LAH, diisobutylaluminum hydride (DIBAL) or the like, to obtain alcohol (13), followed by reacting it with a halogenizing agent such as thionyl chloride or the like, to obtain halide (14), reacting the halide with a sodium alkoxide to obtain ether (15) and reacting it with oxalyl chloride, to obtain an acid chloride; and hydrolyzing it to obtain a carboxylic derivative example (16).

Among the raw materials, the phenol derivative can be prepared for example according to the following processes:





Namely, a substituted phenol (17), is converted into an ether substance (18) by means of tetrahydropyranyl group or the like, followed by reacting it with an organolithium reagent such as butyllithium or the like and iodine, to obtain an iodine substance (19), cyanogenating it, and deprotecting, to obtain a phenol derivative example (20).

Further, the above iodine derivative (19) is reacted with sodium trifluoroacetate/copper iodide (G.E. Carr et al, Journal of the Chemical Society Parkin Trans, Reactions I, 921 (1988)), or methyl fluorosulfonyldifluoroacetate/copper

iodine (Q. Y. Chen et al, Journal of the Chemical Society, Chemical Communications, 705 (1989), to obtain a trifluoromethyl substance (21), followed by deprotecting it, to obtain a phenol derivative example (22).

Further, the above ether substance (18) is reacted with an organolithium reagent such as butyllithium or the like and formylpiperidine, to obtain an aldehyde substance (23), followed by reacting it with a fluorinating agent such as diethylaminosulfurtrifluoride (DAST) (W.J. Middleton et al, Journal of the Organic Chemistry, 40, 574 (1975), S. Rozen et al, Tetrahedron Letters, 41, 111 (1985), M. Hudlicky, Organic Reactions, 35, 513 (1988), P.A. Messina et al, Journal of Fluorine Chemistry, 42, 137 (1989)), to obtain a compound (24), and deprotecting it, to obtain a phenol derivative example (25).

Further, the above aldehyde substance (23) is reduced with a reducing agent such as sodium boron hydride (SBH), lithium aluminum hydride, diisobutylaluminum hydride, or bis(2-methoxyethoxy aluminum sodium hydride or the like, followed by reacting with a fluorinating agent such as DAST or the like, to obtain a compound (26), and deprotecting it, to obtain a phenol derivative example (27).

Further, another substituted phenol substance (28) is treated in the presence of nitric acid and sulfuric acid to obtain a nitro compound (29), followed by converting it into a xanthate according to Albert et al's method (Synthetic Communications, 19, 547-552, 1989), fluorinating it according to Kuroboshi et al's method (Tetrahedron Letters, 33, 29, 4173-4176, 1992), catalytically hydrogenating in the presence of Pt catalyst, to obtain a compound (30), reacting it with hydrochloric acid and sodium nitrite, and hydrolyzing the resulting diazonium salt, to obtain a phenol derivative example (33).

Further, the above nitro compound (29) is reacted in a system of chlorodifluoromethane/sodium hydroxide (Japanese patent application Hyo No. Hei 4-501575), for fluorination, followed by catalytically hydrogenating in the presence of Pt catalyst, to obtain a compound (32), reacting it with hydrochloric acid and sodium nitrite, and hydrolyzing the resulting diazonium salt, to obtain a phenol derivative example (33).

Either of the thus obtained liquid crystalline compound of the present invention exhibit a large $\Delta\epsilon$, and have a small change in the threshold voltage depending on temperatures, are easily blended with various liquid crystal materials, and have a good solubility therewith even at low temperatures; hence the compound is far superior as a constituting component of nematic liquid crystal compositions.

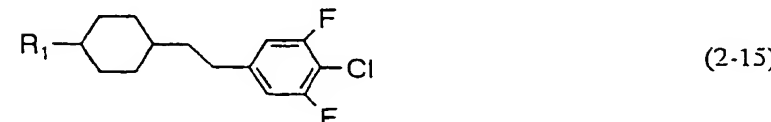
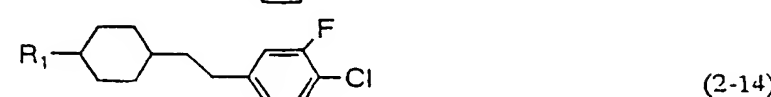
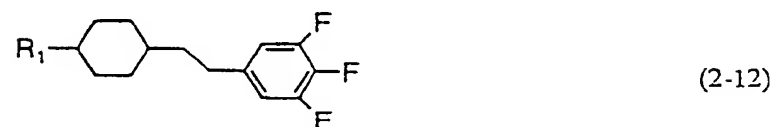
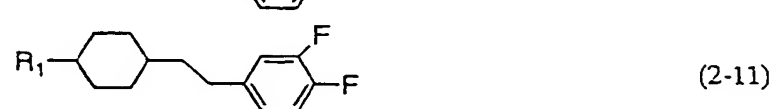
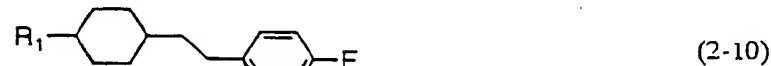
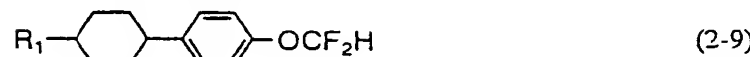
The compound of the present invention is usable as a constituting component in either of liquid crystal compositions of TFT mode, TN mode and STN mode which at present constituted main current, and compounds of $X=CN$ in the formula (I) are particularly preferred for STN, and compounds having X replaced by fluorine are particularly preferred for TFT.

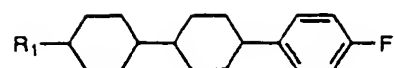
When the compound of the present invention is used as a constituting component of nematic liquid crystal compositions, compounds having a structure of two rings exhibit a relatively low liquid crystal temperature range and a low viscosity, and compounds having a structure of 3 or 4 rings exhibit a broad liquid crystal temperature range and a relatively high viscosity; hence when these compounds are adequately selected, it is possible to obtain liquid crystal compositions having desired physical properties.

Further, the compounds among those of the present invention having a 4-substituted phenyl group, a 3-fluoro-4-substituted phenyl group or 3,5-difluoro-4-substituted phenyl group exhibit a large $\Delta\epsilon$, and when hydrogen atoms in other ring structure are replaced by fluorine atoms, it is possible to impart a further large $\Delta\epsilon$.

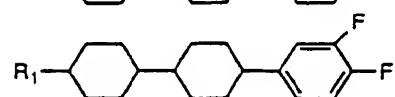
The liquid crystal composition provided by the present invention may be composed only of a first component containing at least one member of the liquid crystalline compound expressed by the formula (1), but the composition is preferred to blend therewith, at least one member of compounds selected from the group consisting of those expressed by the above formulas (2), (3) and (4) (hereinafter abbreviated to the second A component), and/or at least one member of compounds selected from the group consisting of those expressed by the formulas (5), (6), (7), (8) and (9) (hereinafter abbreviated to the second B component), and further, it is possible to additionally blend known compounds as the third component, in order to adjust the threshold voltage, liquid crystal phase temperature range, optical anisotropy value, dielectric anisotropy value, viscosity, etc.

As preferred examples of compounds contained in those expressed by the formulas (2), (3) and (4), among the second A component, the following compounds of (2-1) to (2-15), (3-1) to (3-48) and (4-1) to (4-55) can be respectively mentioned:

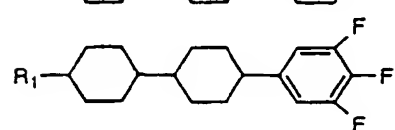




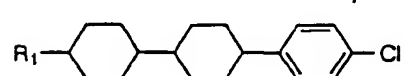
(3-1)



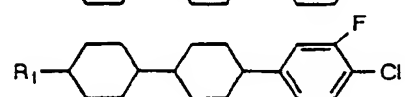
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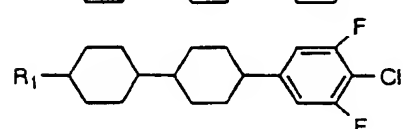
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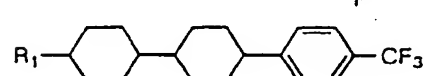
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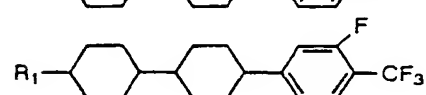
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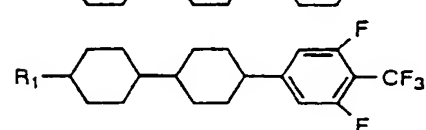
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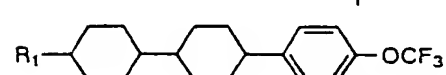
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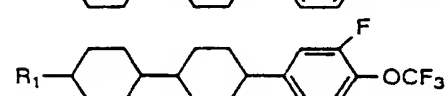
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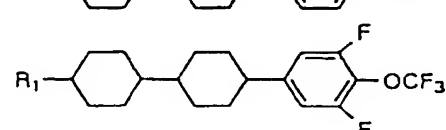
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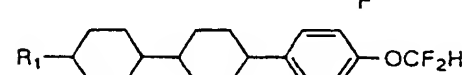
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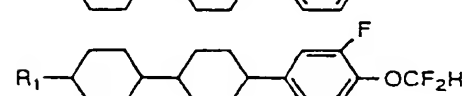
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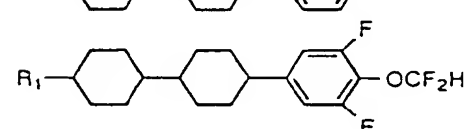
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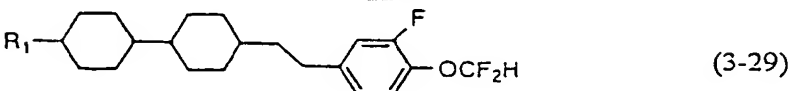
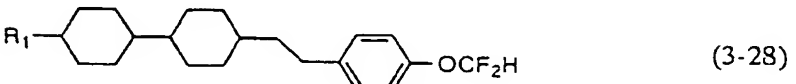
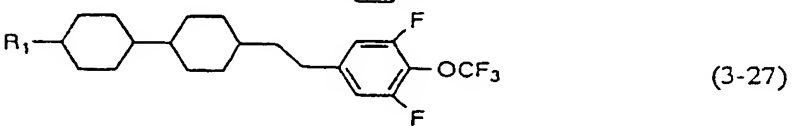
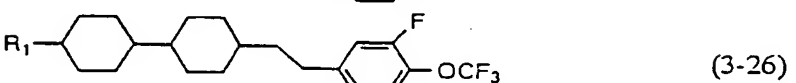
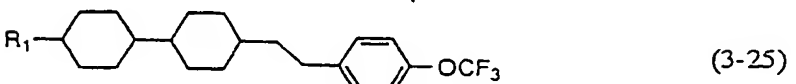
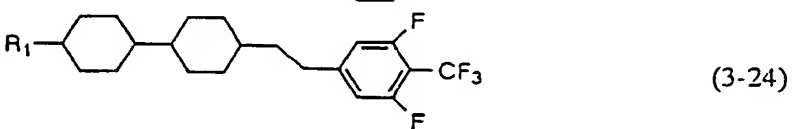
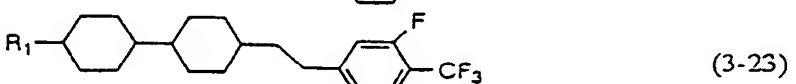
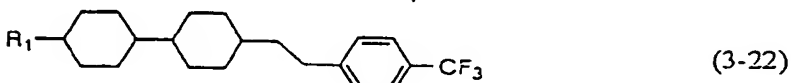
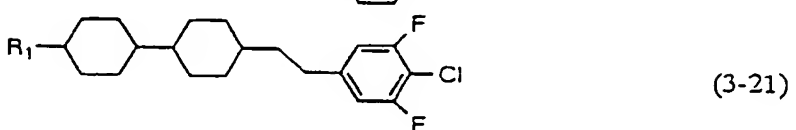
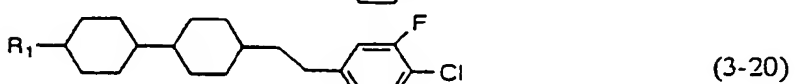
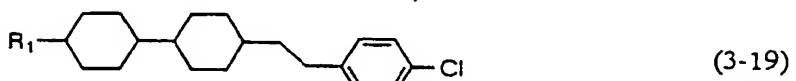
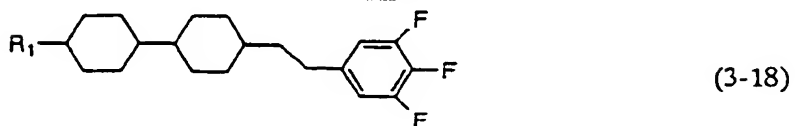
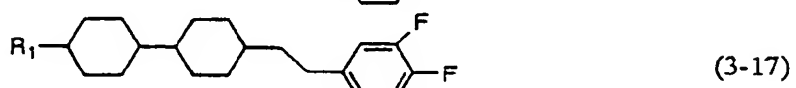
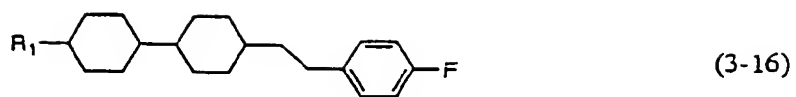
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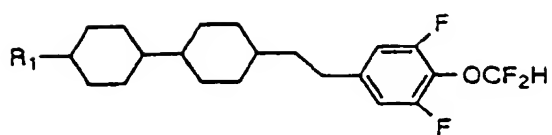


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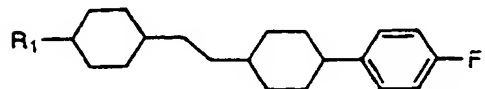


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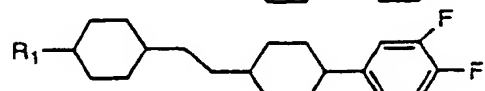




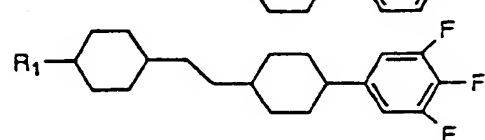
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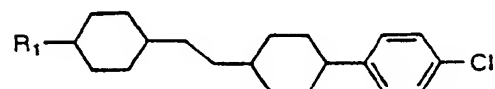
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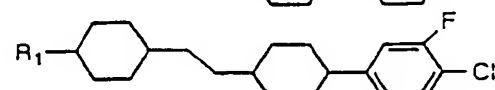
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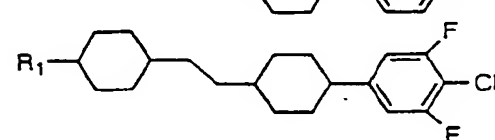
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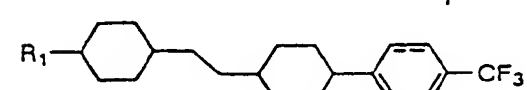
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(3-35)



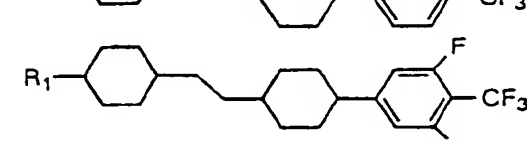
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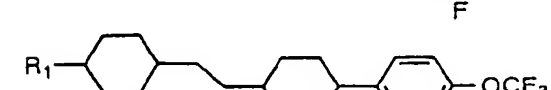
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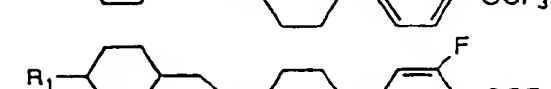
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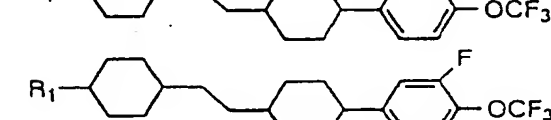
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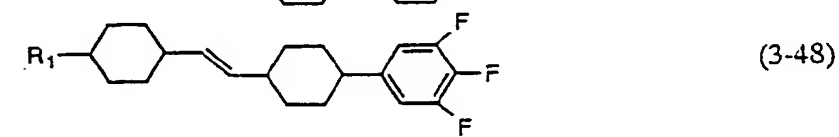
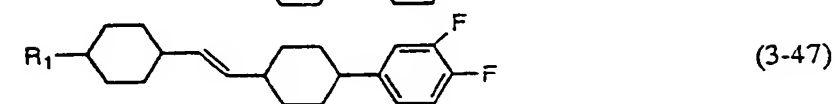
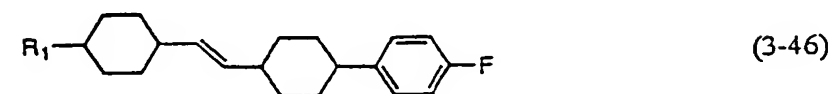
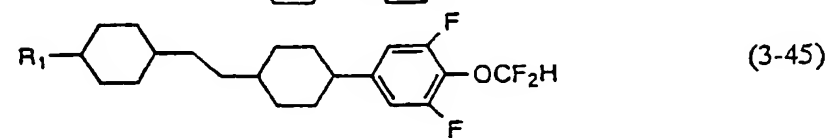
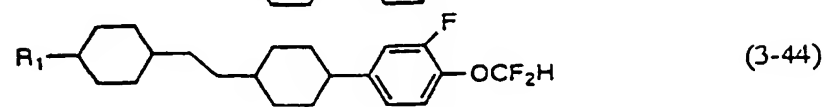
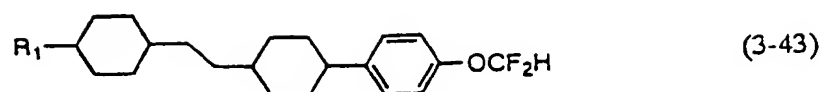


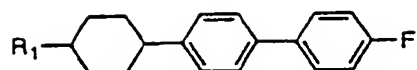
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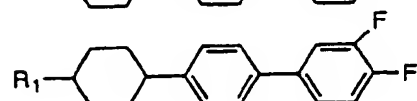
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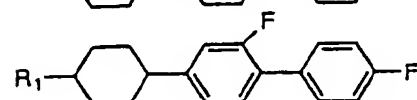




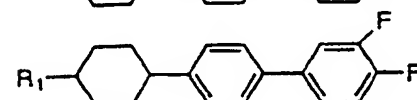
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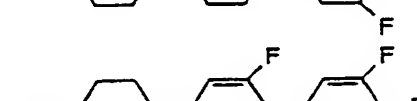
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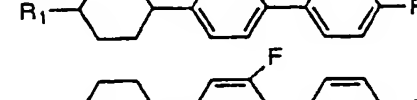
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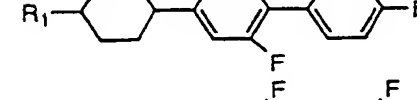
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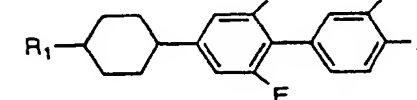
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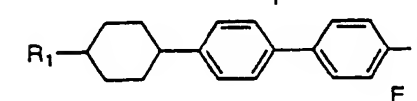
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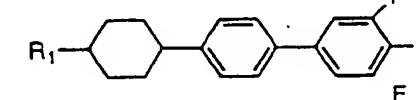
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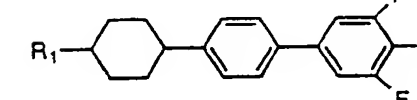
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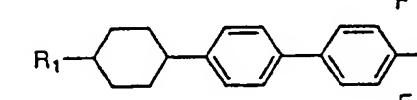
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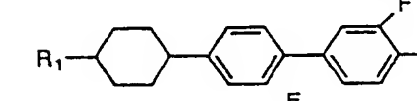
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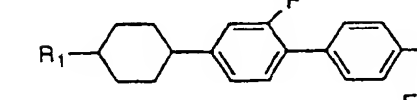
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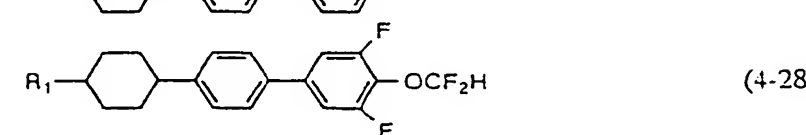
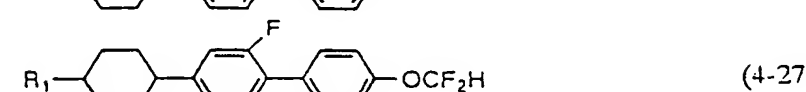
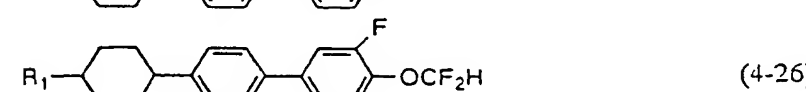
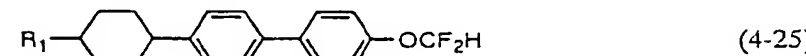
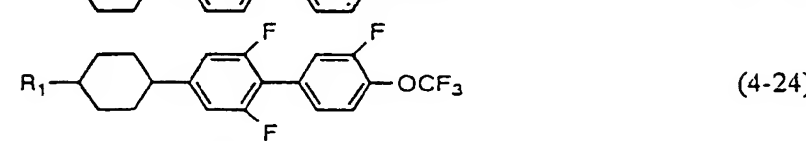
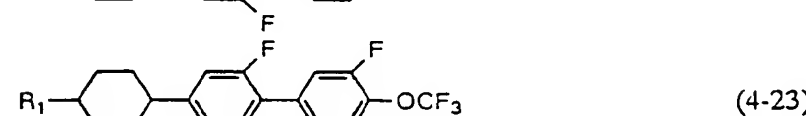
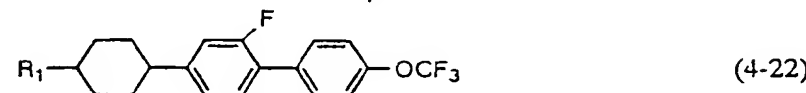
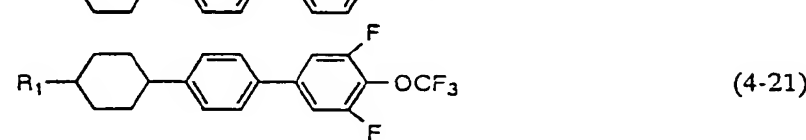
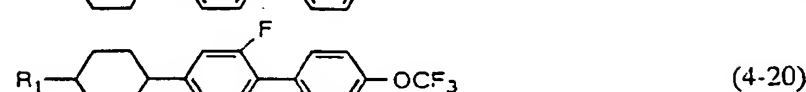
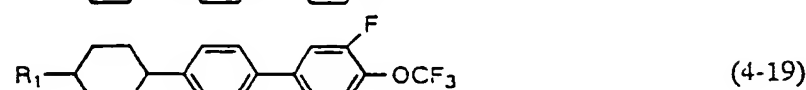
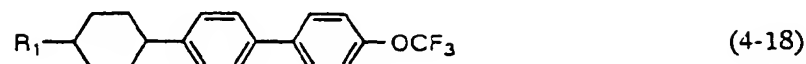
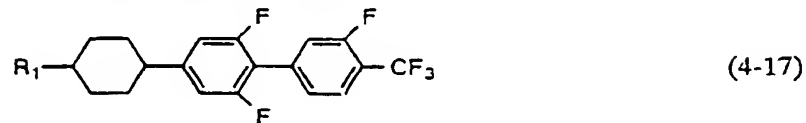
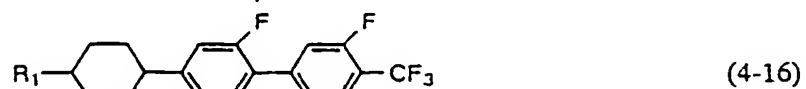
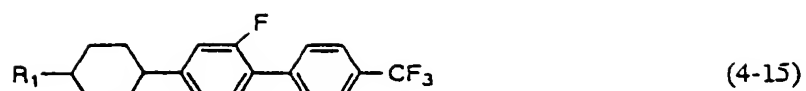
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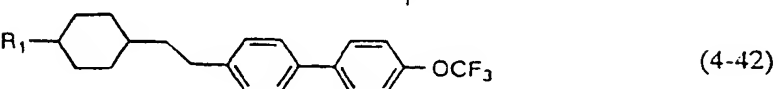
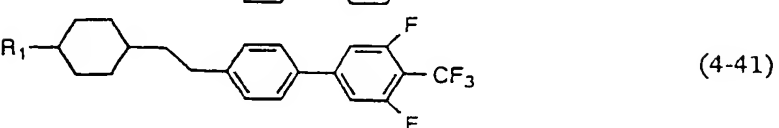
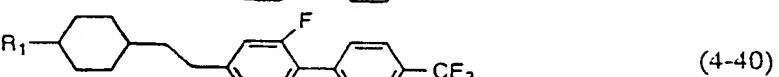
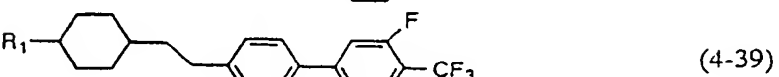
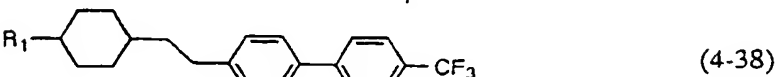
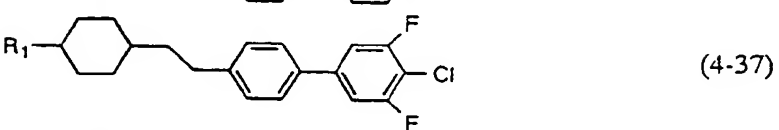
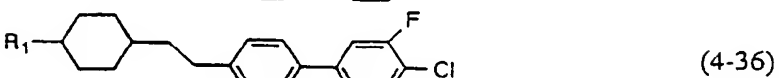
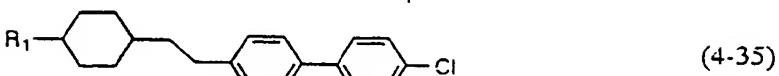
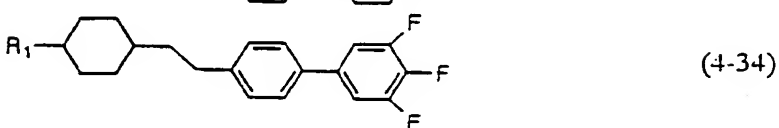
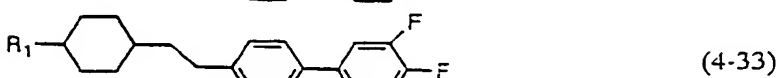
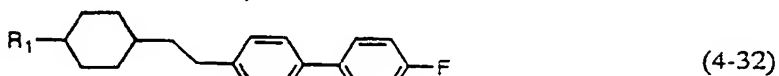
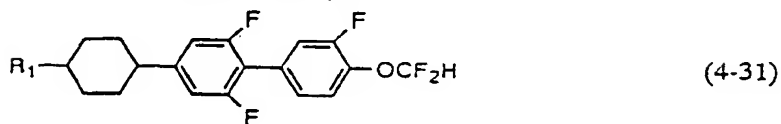
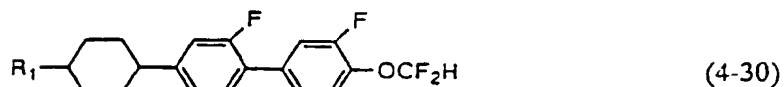
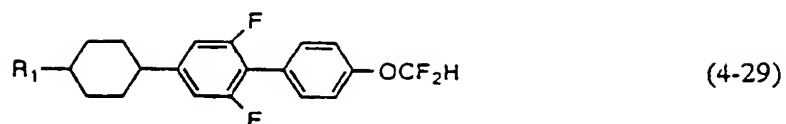


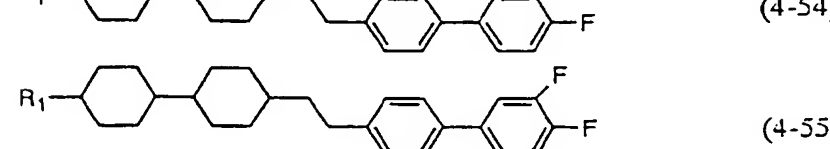
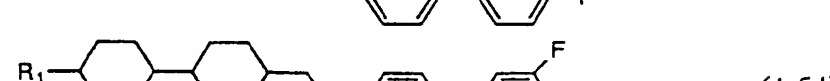
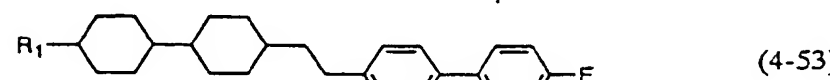
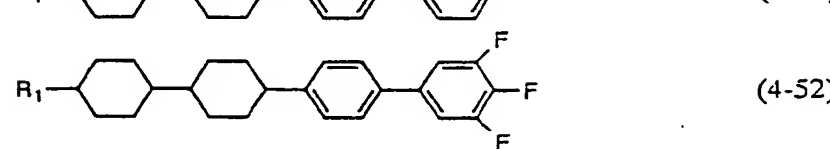
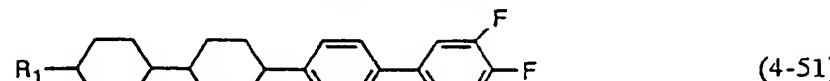
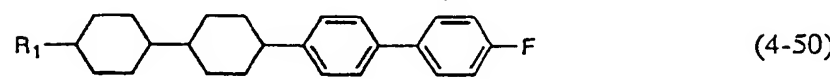
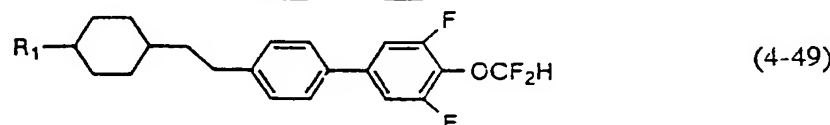
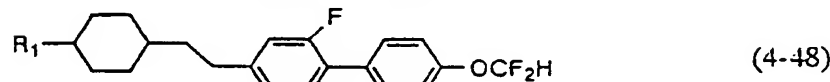
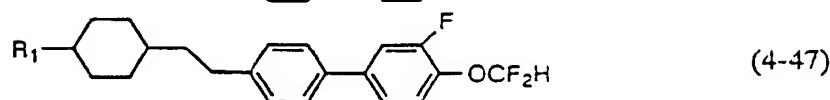
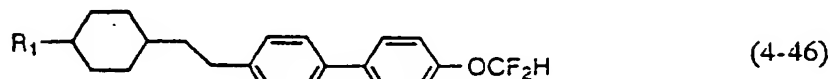
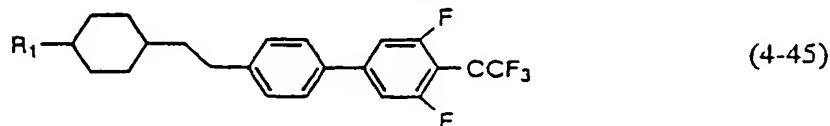
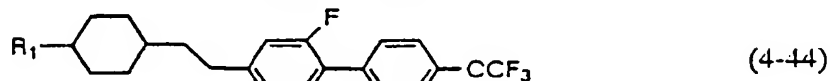
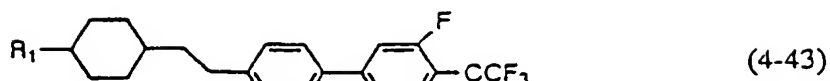
(4-13)



(4-14)



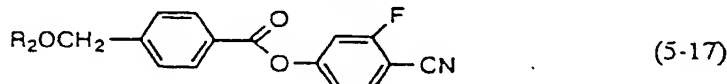
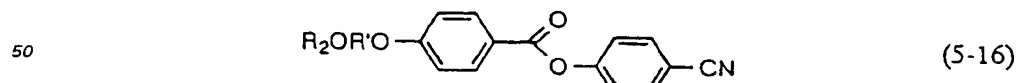
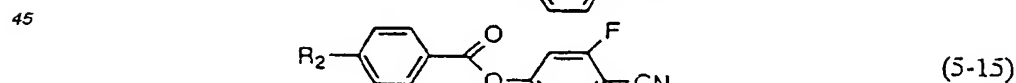
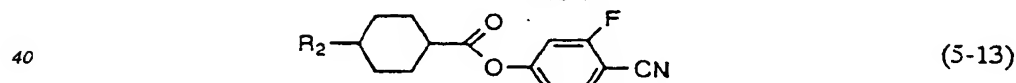
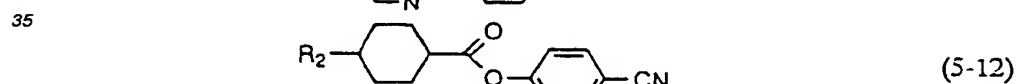
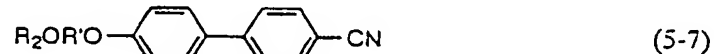
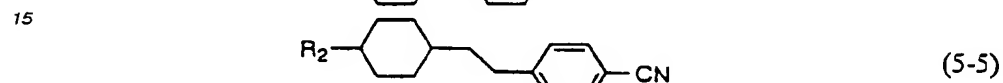
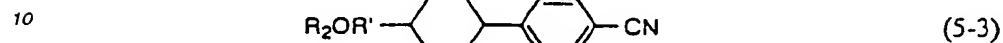




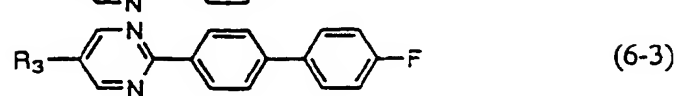
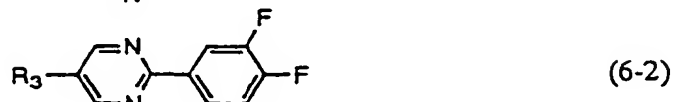
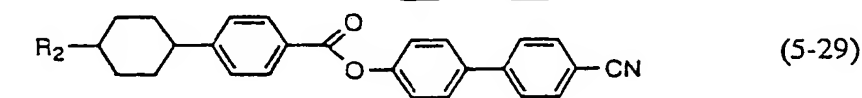
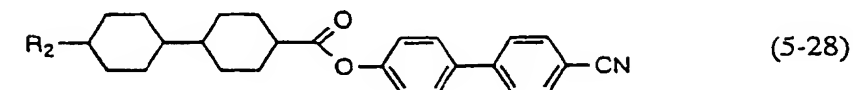
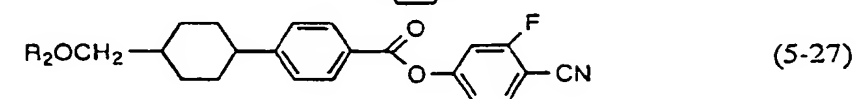
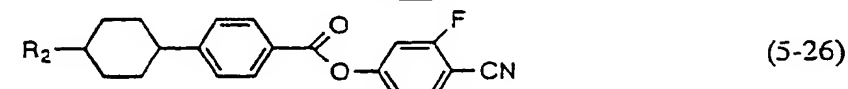
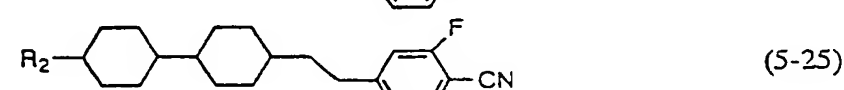
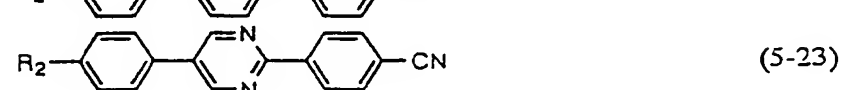
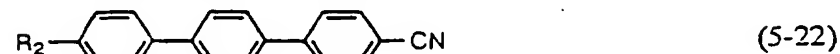
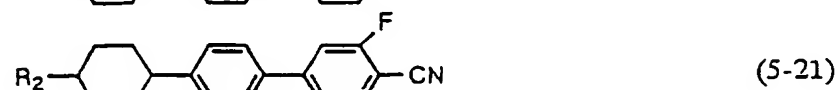
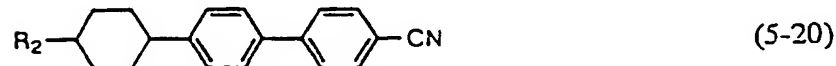
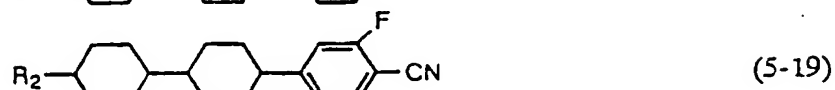
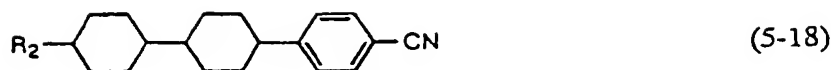
55 The compounds expressed by these formulas (2) to (4) exhibit a positive dielectric anisotropy value and far superior thermal stability and chemical stability.

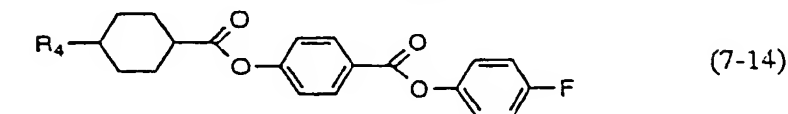
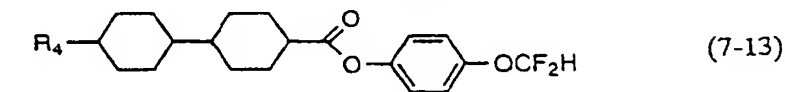
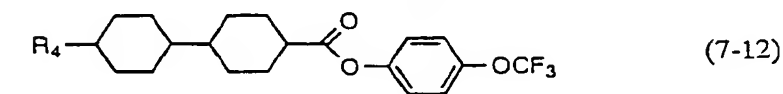
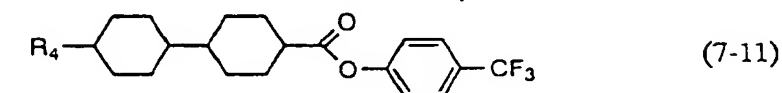
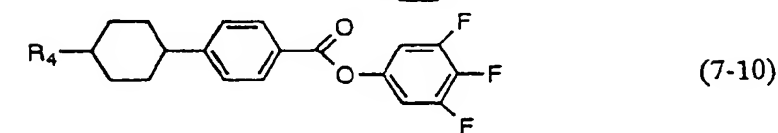
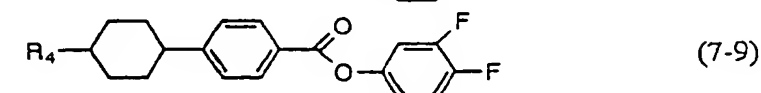
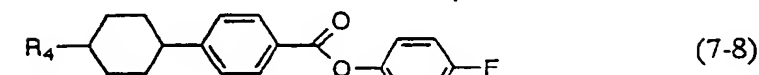
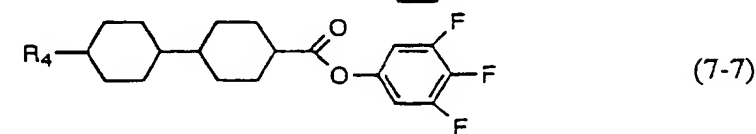
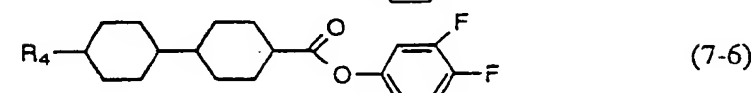
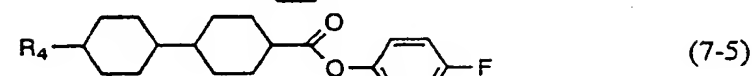
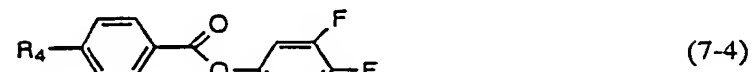
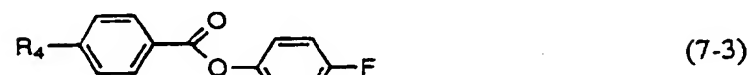
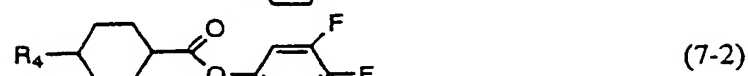
As to the quantity of the compounds used, a range of 1 to 99% by weight based upon the total weight is suitable, and that of 10 to 97% by weight is preferred, and that of 40 to 95% by weight is more preferred.

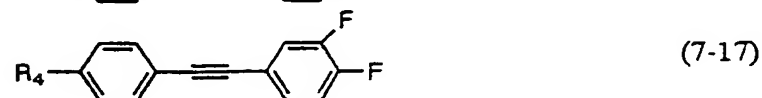
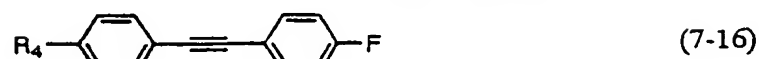
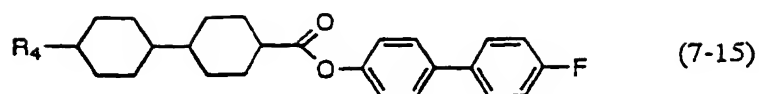
Next, as preferred examples of the compounds expressed by the formulas (5), (6) and (7), among the above second B component, the following (5-1) to (5-29), (6-1) to (6-3) and (7-1) to (7-17) can be mentioned:



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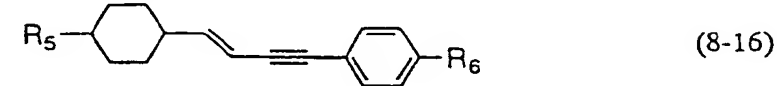
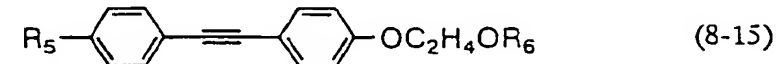
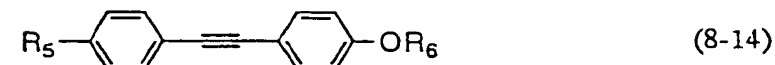
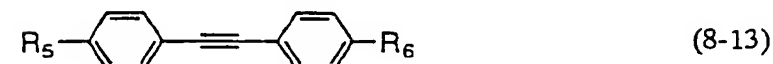
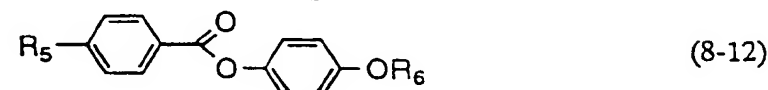
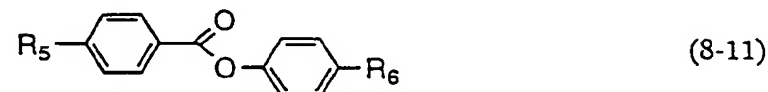
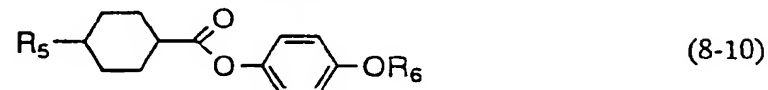
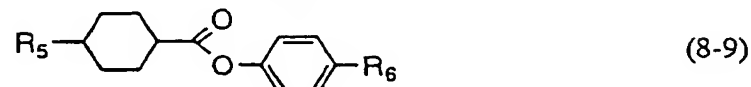
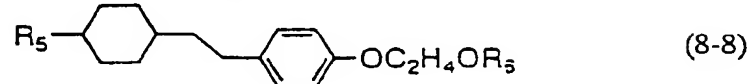
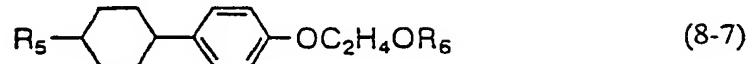
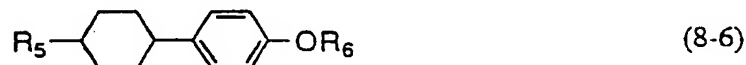
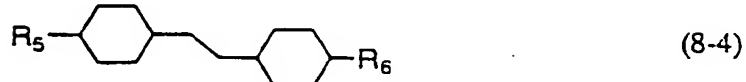
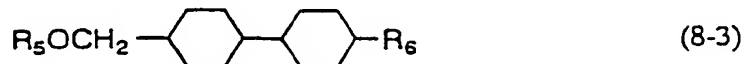


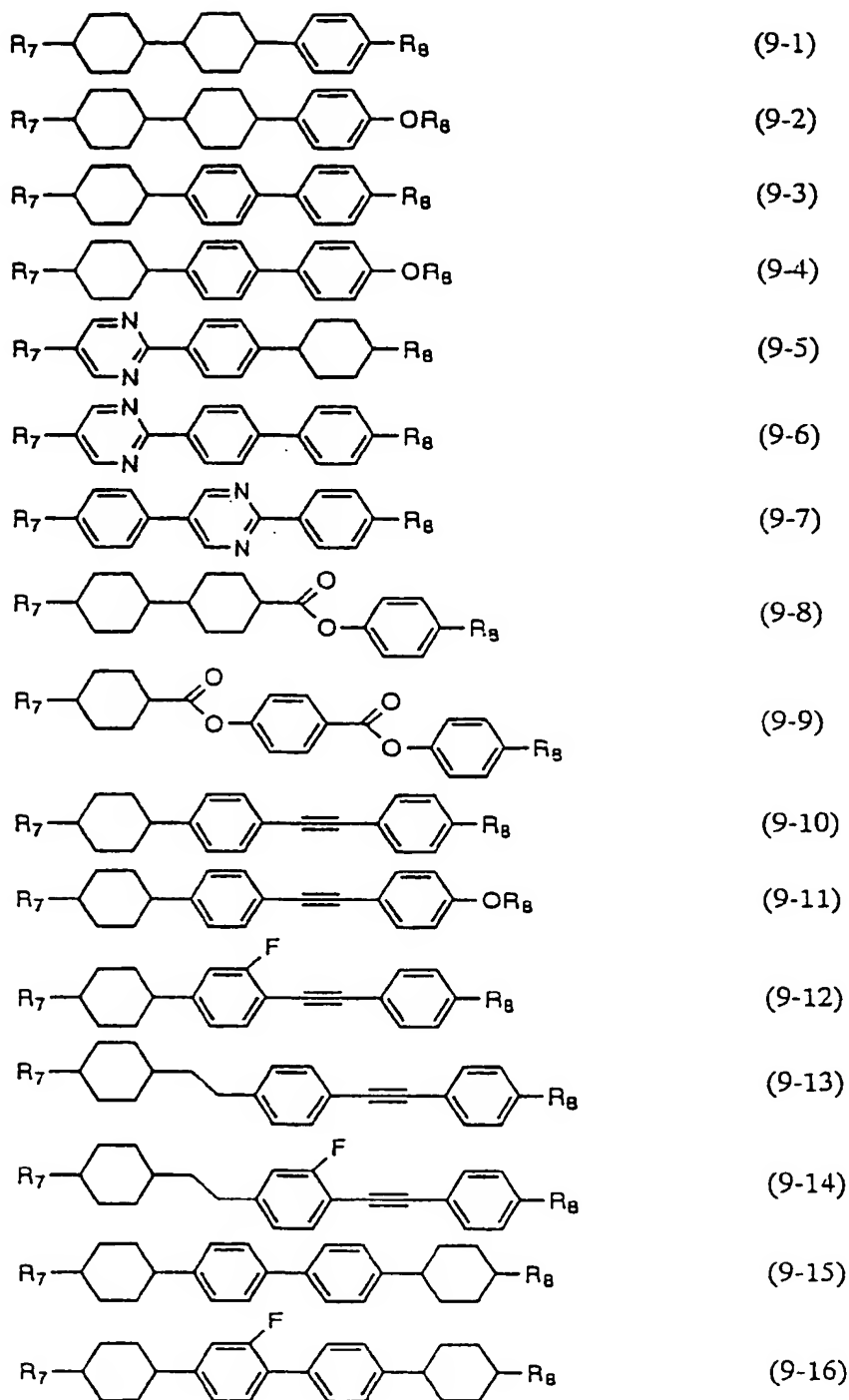




15 The compounds expressed by the formulas (5) to (7) exhibit a positive and large dielectric anisotropy value and are used as composition component, particularly in order to reduce the threshold voltage. Further, the compounds are used for adjusting the viscosity and the optical anisotropy value and for broadening the liquid crystal phase temperature range, and further for improving the steepness.

20 Further, as preferable examples of compounds included in those of the formulas (8) and (9), among those of the second B component, the following (8-1) to (8-16) and (9-1) to (9-16) can be mentioned:





The compounds expressed by the formulas (8) and (9) exhibit a negative or weakly positive dielectric anisotropy value, and among these compounds, those expressed by the formula (8) are used as a composition component, mainly for lowering the viscosity and adjusting the optical anisotropy value, and those expressed by the formula (9) are used for broadening the liquid crystal phase temperature range and/or for adjusting the optical anisotropy value.

The compounds expressed by the formulas (5) to (9) are indispensable for preparing liquid crystal compositions, particularly for STN type display mode or usual TN type display mode. As to the quantity of these compounds used,

when liquid crystal compositions for usual STN type display mode or TN type display mode are prepared, a range of 1 to 99% by weight based upon the total quantity of the liquid crystal compositions is suitable, and a range of 10 to 97% is preferred and that of 40 to 95% by weight is more preferred.

The liquid crystal composition provided by the present invention is preferred to contain at least one member of the liquid crystal compound expressed by the formula (1) in a proportion of 0.1 to 99% by weight, for exhibiting superior characteristics.










































The liquid crystal composition is generally prepared according to a known process, for example according to a process wherein various components are mutually dissolved at high temperatures. Further, if necessary, by adding a suitable additive, improvement is made depending upon the aimed use applications and the composition is optimized. Such additives are well known by person of ordinary skill in the art and described in literatures, etc. in details. Usually, a chiral dopant having an effect of inducing the helical structure to thereby adjust a necessary twist angle and prevent the reverse twist, is added.

Further, when a dichroic pigment such as those of mellocyanine group, styryl group, azo group, azomethine group, azoxy group, quinophthalone group, anthraquinone group, tetrazine group, etc. is added, the resulting composition can be also used as that of GH mode. The composition of the present invention is usable for NCAP prepared by microcapsulating nematic liquid crystals, polymer-dispersion type, liquid crystal display element prepared by forming three-dimensional, reticulated high molecules in liquid crystals (PDLCD), for example, polymer network liquid crystal display element (PNLCD) and besides, as liquid crystal compositions for birefringence control (ECB) mode or DS mode.


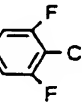

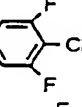

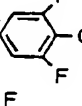

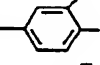


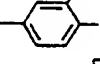


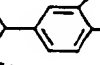








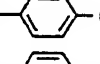


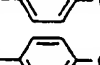


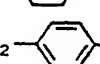

























As liquid crystal composition examples containing the compound of the present invention, the following ones can be mentioned:

In addition, the numbers of the compounds are the same as those shown in the below-mentioned Examples.


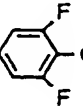

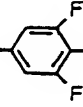


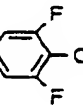

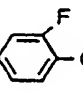

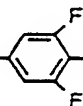































Composition example 1

5		
	$C_3H_7OCH_2$ —  —COO—  —CN (N o. 4)	4 wt. %
10	$C_4H_9OCH_2$ —  —COO—  —CN (N o. 5)	4 wt. %
15	$C_5H_{11}OCH_2$ —  —COO—  —CN (N o. 1)	4 wt. %
20	C_2H_5 —  —  — 	6 wt. %
	C_3H_7 —  —  — 	6 wt. %
25	C_5H_{11} —  —  — 	6 wt. %
	C_3H_7 —  —  —COO— 	5 wt. %
30	C_5H_{11} —  —  —COO— 	5 wt. %
	C_3H_7 —  —COO— 	4 wt. %
35	C_3H_7 —  —  —COOCH ₃	12 wt. %
	C_3H_7 —  —COO—  —OC ₄ H ₉	10 wt. %
40	C_2H_5 —  —  —  —C ₃ H ₇	6 wt. %
	C_3H_7 —  —  —  —C ₃ H ₇	5 wt. %
45	C_4H_9 —  —  —  —C ₃ H ₇	5 wt. %
	C_3H_7 —  —  —OC ₂ H ₅	10 wt. %
50	C_3H_7 —  —  —  —CH ₃	8 wt. %
55		

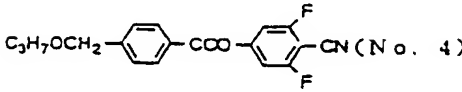
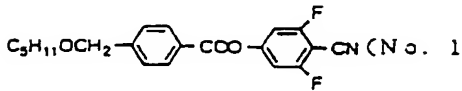
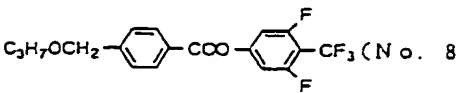
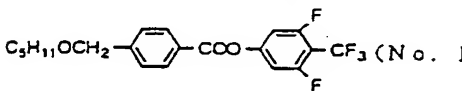
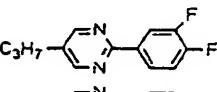
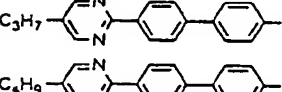
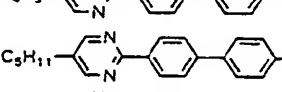
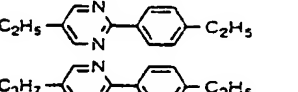
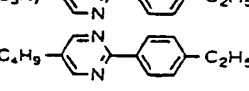
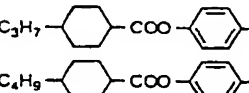
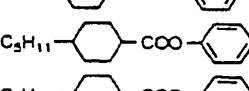
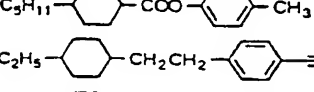
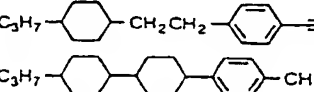
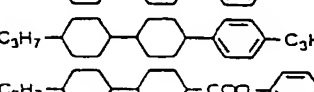
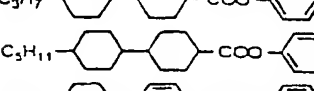
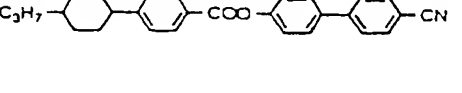




Composition example 2

5	$C_3H_7OCH_2$ —  —COO—  —CF ₃ (No. 8)	5 wt. %
10	$C_4H_9OCH_2$ —  —COO—  —CF ₃ (No. 9)	5 wt. %
15	$C_5H_{11}OCH_2$ —  —COO—  —CF ₃ (No. 10)	5 wt. %
20	C_2H_5 —  —  — 	5 wt. %
25	C_3H_7 —  —  — 	5 wt. %
30	C_5H_{11} —  —  — 	5 wt. %
35	C_2H_5 —  —  —CN	10 wt. %
40	C_3H_7 —  —  —CN	12 wt. %
45	C_5H_{11} —  —  —CN	11 wt. %
50	C_3H_7 —  —  —  —CH ₃	8 wt. %
55	C_3H_7 —  —  —  —C ₃ H ₇	4 wt. %
60	C_3H_7 —  —  —  —OCH ₃	4 wt. %
65	C_3H_7 —  —CH ₂ CH ₂ —  —  —  —C ₂ H ₅	3 wt. %
70	C_3H_7 —  —CH ₂ CH ₂ —  —  —  —C ₃ H ₇	3 wt. %
75	C_3H_7 —  —CH ₂ CH ₂ —  —  —  —C ₄ H ₉	3 wt. %
80	C_3H_7 —  —  —  —  —C ₂ H ₅	4 wt. %
85	C_3H_7 —  —  —  —  —C ₃ H ₇	4 wt. %
90	C_3H_7 —  —  —  —  —C ₄ H ₉	4 wt. %

Composition example 3

5	$\text{C}_3\text{H}_7\text{OCH}_2$ —  — COO —  — CN (N o. 4)	10 wt. %
10	$\text{C}_5\text{H}_{11}\text{OCH}_2$ —  — COO —  — CN (N o. 1)	5 wt. %
15	$\text{C}_2\text{H}_5\text{OCH}_2$ —  —  — COO —  — CN (N o. 187)	8 wt. %
20	$\text{C}_3\text{H}_7\text{OCH}_2$ —  — COO —  — CN	10 wt. %
	$\text{CH}_3\text{CH}=\text{CHC}_2\text{H}_4$ —  — COO —  — CN	15 wt. %
25	C_3H_7 —  —  — OC_2H_5	8 wt. %
	C_3H_7 —  —  — C_4H_9	5 wt. %
30	C_3H_7 —  —  — COO — 	5 wt. %
	C_5H_{11} —  —  — COO — 	4 wt. %
35	C_3H_7 —  —  — 	6 wt. %
	C_3H_7 —  —  — 	6 wt. %
	C_3H_7 —  —  — 	3 wt. %
40	C_3H_7 —  —  — COO — 	3 wt. %
	C_3H_7 —  — CH_2CH_2 —  — \equiv —  — C_2H_5	4 wt. %
45	C_3H_7 —  — CH_2CH_2 —  — \equiv —  — C_3H_7	4 wt. %
	C_3H_7 —  —  — \equiv —  — C_2H_5	4 wt. %

Composition example 4

5		6 wt. %
10		5 wt. %
		5 wt. %
15		3 wt. %
20		6 wt. %
		4 wt. %
		4 wt. %
25		4 wt. %
		5 wt. %
30		5 wt. %
		5 wt. %
35		5 wt. %
		5 wt. %
		5 wt. %
40		6 wt. %
		4 wt. %
		4 wt. %
45		5 wt. %
		5 wt. %
		3 wt. %
50		3 wt. %
		3 wt. %

Composition example 5

5		8 wt. %
10		5 wt. %
		5 wt. %
15		5 wt. %
20		8 wt. %
		4 wt. %
25		10 wt. %
		3 wt. %
30		4 wt. %
		3 wt. %
35		6 wt. %
		6 wt. %
40		4 wt. %
		5 wt. %
		5 wt. %
45		5 wt. %
		6 wt. %
50		4 wt. %
		4 wt. %

Composition example 6


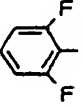


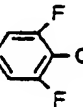



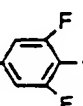
































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10		6 wt. %
		5 wt. %
15		8 wt. %
		10 wt. %
20		8 wt. %
		3 wt. %
		3 wt. %
25		4 wt. %
		5 wt. %
30		3 wt. %
		3 wt. %
35		3 wt. %
		3 wt. %
40		5 wt. %
		6 wt. %
45		5 wt. %
		4 wt. %
50		4 wt. %
		6 wt. %

55

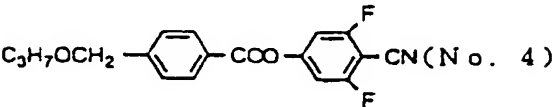
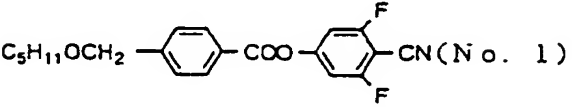
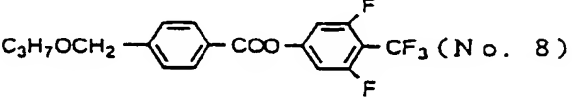
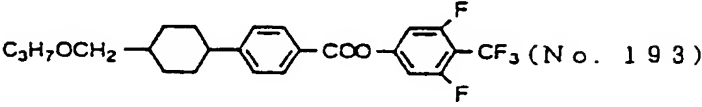
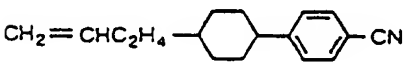
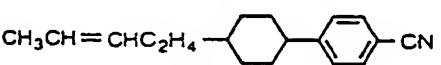
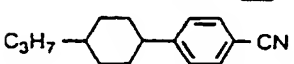
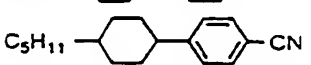
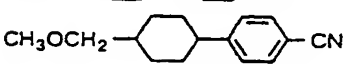
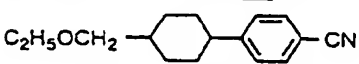
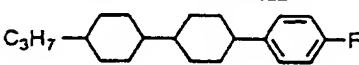
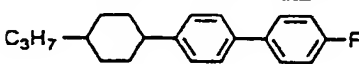
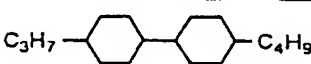
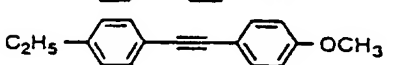
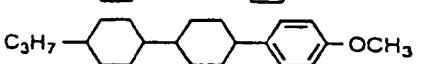
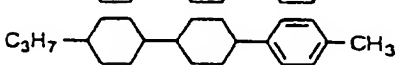
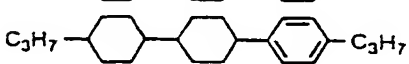
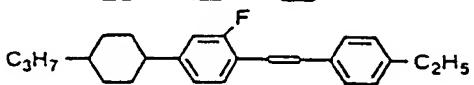
Composition example 7

5		6 wt. %
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15		4 wt. %
20		4 wt. %
25		10 wt. %
		10 wt. %
30		5 wt. %
		5 wt. %
35		6 wt. %
		4 wt. %
40		5 wt. %
		3 wt. %
45		3 wt. %
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		4 wt. %
55		3 wt. %
		6 wt. %
		3 wt. %
		3 wt. %


























































Composition example 8

5	$C_3H_7OCH_2$ —  —COO—  —CN (N o. 4)	10 wt. %
10	$C_2H_5OCH_2$ —  —  —COO—  —CN (N o. 187)	9 wt. %
15	$C_2H_5OCH_2$ —  —  —COO—  —  —CN (N o. 295)	5 wt. %
	C_2H_5 —  —  —CN	5 wt. %
20	C_3H_7 —  —  —CN	15 wt. %
	C_2H_5 —  —  —  —CN	5 wt. %
	C_3H_7 —  —  —  —CN	5 wt. %
25	C_4H_9 —  —  —  —CN	5 wt. %
	C_3H_7 —  —  —  —F	10 wt. %
30	C_3H_7 —  —  — C_4H_9	8 wt. %
	C_5H_{11} —  —  — C_4H_9	3 wt. %
35	C_2H_5 —  — \equiv —  —OCH ₃	4 wt. %
	C_3H_7 —  — \equiv —  —OCH ₃	4 wt. %
40	C_4H_9 —  — \equiv —  —OCH ₃	4 wt. %
	C_3H_7 —  —  —  —CH ₃	5 wt. %
45	C_3H_7 —  —  —  —OCH ₃	3 wt. %

Composition example 9


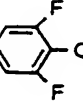


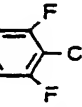


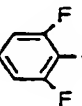















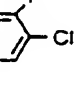


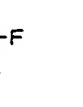


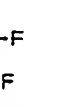


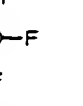


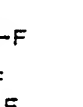


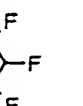





5		6 wt. %
10		6 wt. %
15		3 wt. %
20		3 wt. %
25		8 wt. %
30		8 wt. %
35		15 wt. %
40		8 wt. %
45		4 wt. %
50		4 wt. %
55		2 wt. %
60		2 wt. %
65		6 wt. %
70		6 wt. %
75		3 wt. %
80		5 wt. %
85		8 wt. %
90		3 wt. %

Composition example 10




























































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10	$C_5H_{11}OCH_2$ —  —COO—  — CF_3 (N o. 10)	5 wt. %
	$C_3H_7OCH_2$ —  —COO—  —  — CF_3 (N o. 115)	6 wt. %
15	$C_8H_9OCH_2$ —  —  —COO—  —  — CF_3 (N o. 301)	3 wt. %
	C_7H_{15} —  —  —F	3 wt. %
20	C_5H_{11} —  —CH ₂ CH ₂ —  —F	4 wt. %
	C_2H_5 —  —  —  —F	10 wt. %
25	C_3H_7 —  —  —  —F	10 wt. %
	C_5H_{11} —  —  —  —F	10 wt. %
30	C_2H_5 —  —CH ₂ CH ₂ —  —  —F	4 wt. %
	C_3H_7 —  —CH ₂ CH ₂ —  —  —F	2 wt. %
35	C_5H_{11} —  —CH ₂ CH ₂ —  —  —F	4 wt. %
	C_2H_5 —  —  —  —F	4 wt. %
40	C_3H_7 —  —  —  —F	4 wt. %
	C_5H_{11} —  —  —  —F	8 wt. %
45	C_2H_5 —  —  —  —F	3 wt. %
	C_3H_7 —  —  —  —F	3 wt. %
	C_5H_{11} —  —  —  —F	2 wt. %
50	C_3H_7 —  —  —  —F	4 wt. %
	C_3H_7 —  —  —  —CH ₃	5 wt. %

55


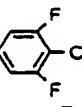

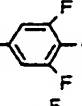

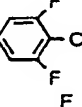

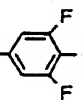













































Composition example 11

5	$C_3H_7OCH_2$ —  —COO—  (N o. 8)	5 wt. %
10	$C_3H_7OCH_2$ —  —  —COO—  (N o. 193)	5 wt. %
15	$C_5H_{11}OCH_2$ —  —  —COO—  (N o. 194)	5 wt. %
	C_3H_7 —  — 	4 wt. %
20	C_5H_{11} —  — 	4 wt. %
	C_2H_5 —  —  — 	5 wt. %
25	C_3H_7 —  —  — 	6 wt. %
	C_5H_{11} —  —  — 	5 wt. %
30	C_2H_5 —  —CH ₂ CH ₂ —  — 	4 wt. %
	C_2H_5 —  —  — 	6 wt. %
35	C_3H_7 —  —  — 	6 wt. %
40	C_5H_{11} —  —  — 	12 wt. %
	C_3H_7 —  —  — 	13 wt. %
45	C_5H_{11} —  —  — 	13 wt. %
50	C_3H_7 —  —  —  —C ₂ H ₅	3 wt. %
	C_3H_7 —  —  —C ₄ H ₉	4 wt. %

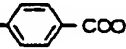







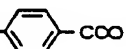



































Composition example 12

5	$C_2H_5OCH_2$ —  —  —COO—  —CN (No. 187)	8 wt. %
10	$C_3H_7OCH_2$ —  —COO—  —  —CN (No. 108)	6 wt. %
	$C_5H_{11}OCH_2$ —  —COO—  —  —CN (No. 110)	6 wt. %
15	C_7H_{15} —  — 	7 wt. %
	C_2H_5 —  —  — 	8 wt. %
20	C_3H_7 —  —  — 	8 wt. %
	C_5H_{11} —  —  — 	8 wt. %
25	C_2H_5 —  —CH ₂ CH ₂ —  — 	6 wt. %
	C_3H_7 —  —CH ₂ CH ₂ —  — 	3 wt. %
30	C_5H_{11} —  —CH ₂ CH ₂ —  — 	6 wt. %
	C_2H_5 —  —  — 	3 wt. %
35	C_3H_7 —  —  — 	3 wt. %
	C_3H_7 —  —  —COO— 	4 wt. %
40	C_3H_7 —  —  —COO— 	3 wt. %
	C_3H_7 —  —  — 	5 wt. %
45	C_3H_7 —  —  —COO— 	3 wt. %
	C_5H_{11} —  —  —COO— 	3 wt. %
	C_3H_7 —  —  —COO— 	3 wt. %
50	C_3H_7 —  —  — 	3 wt. %
	C_3H_7 —  —  — 	4 wt. %

Composition example 13

5	$C_3H_7OCH_2$ —  —COO—  —CN (N o. 4)	3 wt. %
10	$C_5H_{11}OCH_2$ —  —COO—  —CN (N o. 1)	3 wt. %
	$C_3H_7OCH_2$ —  —COO—  —CF ₃ (N o. 8)	5 wt. %
15	$C_5H_{11}OCH_2$ —  —COO—  —CF ₃ (N o. 10)	4 wt. %
20	C_7H_{15} —  —  —F	5 wt. %
	C_2H_5 —  —  —  —F	11 wt. %
25	C_3H_7 —  —  —  —F	11 wt. %
	C_5H_{11} —  —  —  —F	11 wt. %
30	C_2H_5 —  —  —  —F	5 wt. %
	C_3H_7 —  —  —  —F	5 wt. %
35	C_5H_{11} —  —  —  —F	10 wt. %
	C_3H_7 —  —CH ₂ CH ₂ —  —  —F	3 wt. %
40	C_5H_{11} —  —CH ₂ CH ₂ —  —  —F	3 wt. %
	C_3H_7 —  —  —  —F	4 wt. %
45	C_3H_7 —  —  —COO—  —  —F	4 wt. %
	C_5H_{11} —  —  —COO—  —  —F	3 wt. %
	C_3H_7 —  —  —OC ₂ H ₅	3 wt. %
50	CH ₃ OCH ₂ —  —  —C ₃ H ₇	2 wt. %
	C_3H_7 —  —  —  —  —C ₂ H ₅	5 wt. %

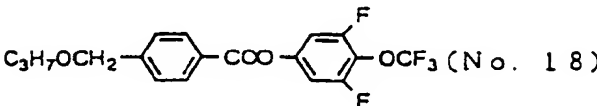
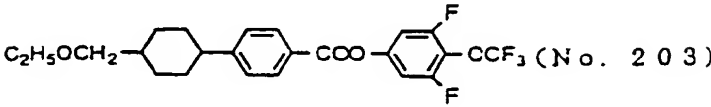
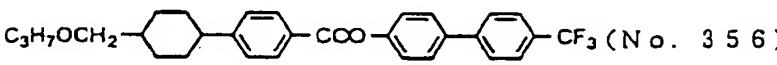
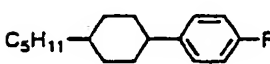
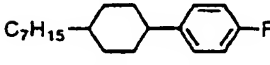
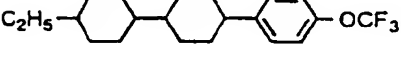
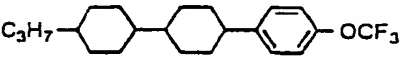
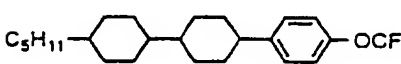
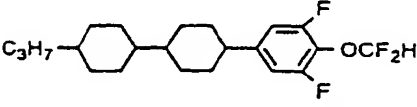
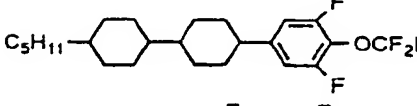
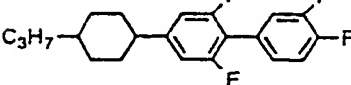
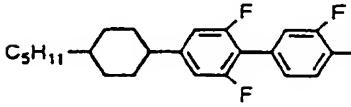
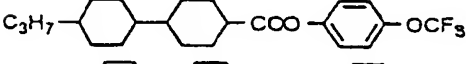
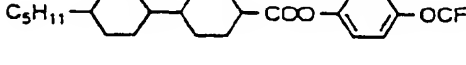
Composition example 14

5	$C_3H_7OCH_2$ —  —COO—  —CF ₃ (N o. 8)	5 wt. %
10	$C_3H_7OCH_2$ —  —COO—  —  —CF ₃ (N o. 1 4 5)	4 wt. %
	$C_5H_{11}OCH_2$ —  —COO—  —  —CF ₃ (N o. 1 4 6)	4 wt. %
15	$C_3H_7OCH_2$ —  —COO—  —  —CF ₃ (N o. 3 4 1)	4 wt. %
	$C_3H_7OCH_2$ —  —  —COO—  —  —CF ₃ (N o. 3 5 6)	2 wt. %
20	C_7H_{15} —  —  —F	7 wt. %
	C_3H_7 —  —  —  —F	8 wt. %
25	C_4H_9 —  —  —  —F	4 wt. %
	C_3H_7 —  —CH ₂ CH ₂ —  —  —F	10 wt. %
30	C_4H_9 —  —CH ₂ CH ₂ —  —  —F	8 wt. %
35	C_5H_{11} —  —CH ₂ CH ₂ —  —  —F	8 wt. %
40	C_3H_7 —  —  —CH ₂ CH ₂ —  —F	12 wt. %
45	C_5H_{11} —  —  —CH ₂ CH ₂ —  —F	8 wt. %
	C_3H_7 —  —  —  —F	8 wt. %
50	C_5H_{11} —  —  —  —F	8 wt. %

Composition example 15

5		8 wt. %
10		6 wt. %
15		5 wt. %
20		2 wt. %
25		2 wt. %
30		10 wt. %
35		8 wt. %
40		8 wt. %
45		5 wt. %
50		5 wt. %
55		5 wt. %
		5 wt. %
		4 wt. %
		4 wt. %
		10 wt. %
		8 wt. %
		3 wt. %
		2 wt. %

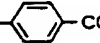



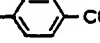















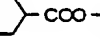
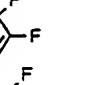

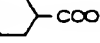
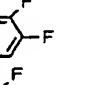

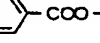
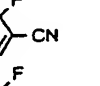

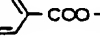
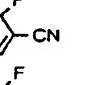

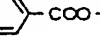
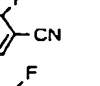

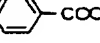
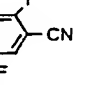



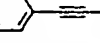



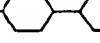

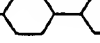

Composition example 16

5		10 wt. %
10		6 wt. %
15		5 wt. %
		12 wt. %
20		12 wt. %
		5 wt. %
25		5 wt. %
		5 wt. %
30		12 wt. %
35		10 wt. %
		5 wt. %
40		5 wt. %
45		4 wt. %
		4 wt. %

Composition example 17

5		5 wt. %
10		5 wt. %
		5 wt. %
15		5 wt. %
20		3 wt. %
25		4 wt. %
		3 wt. %
30		6 wt. %
		5 wt. %
35		6 wt. %
		4 wt. %
40		6 wt. %
		5 wt. %
45		6 wt. %
		6 wt. %
50		4 wt. %
		3 wt. %
55		3 wt. %
		3 wt. %
		3 wt. %
		6 wt. %
		4 wt. %

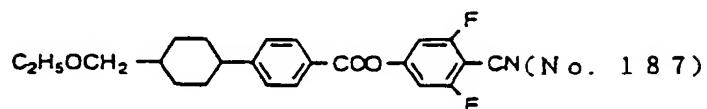
Composition example 18

5	$C_3H_7OCH_2$ —  —COO—  —CF ₃ (No. 8)	8 wt. %
10	$C_5H_{11}OCH_2$ —  —COO—  —CF ₃ (No. 10)	8 wt. %
	$C_3H_7OCH_2$ —  —COO—  —  —CF ₃ (No. 115)	5 wt. %
15	C_4H_9 —  —COO—  —F	5 wt. %
	C_5H_{11} —  —COO—  —F	5 wt. %
20	C_2H_5 —  —COO—  —CN	6 wt. %
	C_3H_7 —  —COO—  —CN	6 wt. %
25	C_6H_9 —  —COO—  —CN	5 wt. %
	C_5H_{11} —  —COO—  —CN	5 wt. %
30	C_3H_7 —  —  —COO—  —F	4 wt. %
	C_5H_{11} —  —  —COO—  —F	4 wt. %
35	C_2H_5 —  —  —COO—  —CN	6 wt. %
	C_3H_7 —  —  —COO—  —CN	6 wt. %
40	C_4H_9 —  —  —COO—  —CN	4 wt. %
	C_5H_{11} —  —  —COO—  —CN	4 wt. %
45	$CH_3OC_3H_8$ —  —COO—  —CN	5 wt. %
	C_3H_7 —  —  —  —  —C ₂ H ₅	8 wt. %
50	$CH_2=CHC_2H_4$ —  —  —C ₃ H ₇	3 wt. %
	$CH_2=CHC_2H_4$ —  —  —  —CH ₃	3 wt. %

55

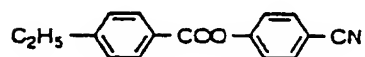
Composition example 19

5



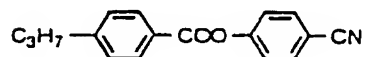
12 wt. %

10



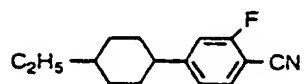
12 wt. %

15

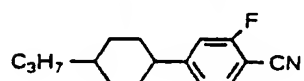


3 wt. %

20

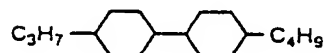


10 wt. %

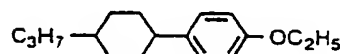


7 wt. %

25

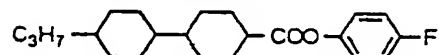


9 wt. %

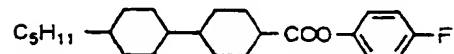


6 wt. %

30

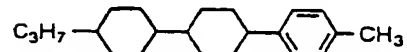


5 wt. %

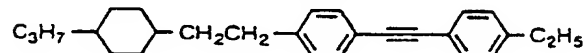


5 wt. %

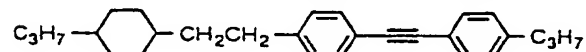
35



9 wt. %

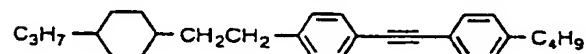


5 wt. %

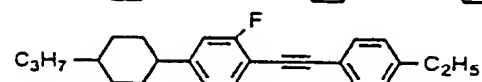


5 wt. %

40



5 wt. %




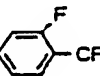






























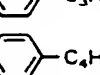





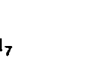









7 wt. %

45




























































50

55

Composition example 20

5	$C_3H_7OCH_2$ —  —COO—  (N o. 3 3 8)	5 wt. %
10	$C_4H_9OC_2H_4$ —  —COO—  (N o. 7 2)	5 wt. %
15	$C_5H_{11}OCH_2$ —  —COO—  (N o. 2 0)	5 wt. %
20	C_2H_5 —  —  —  (N o. 3 3 8)	5 wt. %
25	C_3H_7 —  —  —  (N o. 7 2)	5 wt. %
30	C_5H_{11} —  —  —  (N o. 2 0)	5 wt. %
35	C_2H_5 —  —  (N o. 3 3 8)	10 wt. %
40	C_3H_7 —  —  (N o. 7 2)	12 wt. %
45	C_5H_{11} —  —  (N o. 2 0)	11 wt. %
50	C_3H_7 —  —  —  (N o. 3 3 8)	8 wt. %
55	C_3H_7 —  —  —  (N o. 7 2)	4 wt. %
60	C_3H_7 —  —  —  (N o. 2 0)	4 wt. %
65	C_3H_7 —  —CH ₂ CH ₂ —  ≡  —C ₂ H ₅	3 wt. %
70	C_3H_7 —  —CH ₂ CH ₂ —  ≡  —C ₃ H ₇	3 wt. %
75	C_3H_7 —  —CH ₂ CH ₂ —  ≡  —C ₄ H ₉	3 wt. %
80	C_3H_7 —  —  ≡  —C ₂ H ₅	4 wt. %
85	C_3H_7 —  —  ≡  —C ₃ H ₇	4 wt. %
90	C_3H_7 —  —  ≡  —C ₄ H ₉	4 wt. %

Composition example 21

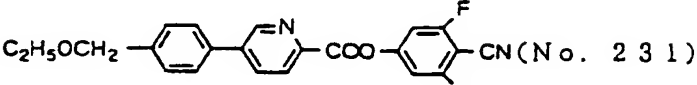
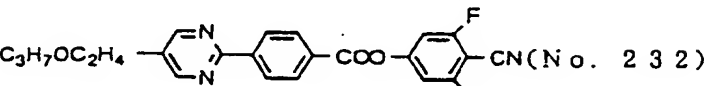
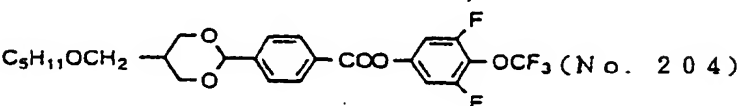
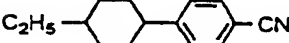
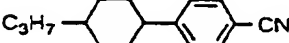
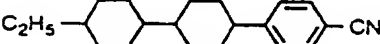
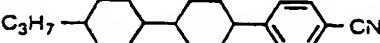
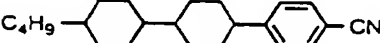
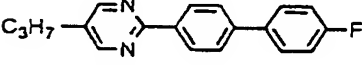
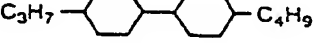
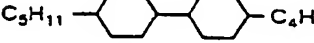
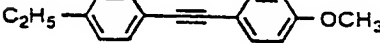
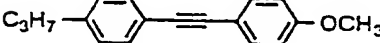
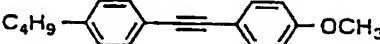


5	$C_3H_7OCH_2$ -  -COO-  -CF ₃ (N o. 3 3 5)	6 wt. %
	$C_3H_7OCH_2$ -  -COO-  -OCF ₃ (N o. 1 8)	5 wt. %
10	$C_3H_7OCH_2$ -  -COO-  -CFH ₂ (N o. 2 8)	5 wt. %
15	$C_5H_{11}OCH_2$ -  -COO-  -OCF ₃ (N o. 3 9)	3 wt. %
	C_3H_7 -  - 	6 wt. %
20	C_3H_7 -  -  - 	4 wt. %
	C_4H_9 -  -  - 	4 wt. %
25	C_5H_{11} -  -  - 	4 wt. %
	C_2H_5 -  -  -C ₂ H ₅	5 wt. %
	C_3H_7 -  -  -C ₂ H ₅	5 wt. %
30	C_4H_9 -  -  -C ₂ H ₅	5 wt. %
	C_3H_7 -  -COO-  -OC ₄ H ₉	5 wt. %
35	C_4H_9 -  -COO-  -OC ₂ H ₅	5 wt. %
	C_5H_{11} -  -COO-  -OCH ₃	5 wt. %
	C_5H_{11} -  -COO-  -CH ₃	6 wt. %
40	C_2H_5 -  -CH ₂ CH ₂ -  -  -  -C ₄ H ₉	4 wt. %
	C_3H_7 -  -CH ₂ CH ₂ -  -  -  -C ₄ H ₉	4 wt. %
45	C_3H_7 -  -  -  -CH ₃	5 wt. %
	C_3H_7 -  -  -  -C ₃ H ₇	5 wt. %
	C_3H_7 -  -  -COO-  - 	3 wt. %
50	C_5H_{11} -  -  -COO-  - 	3 wt. %
	C_3H_7 -  -  -COO-  - 	3 wt. %

55

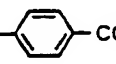
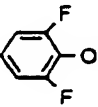


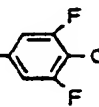
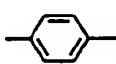
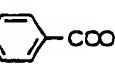
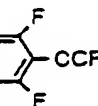

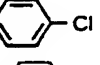

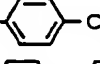











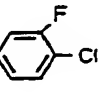

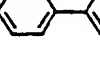
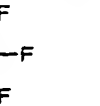

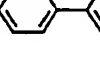
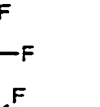

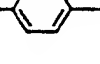
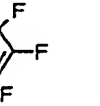

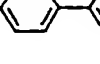
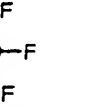

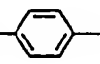
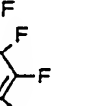

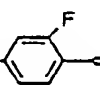




Composition example 22

5		6 wt. %
10		6 wt. %
15		4 wt. %
20		4 wt. %
25		10 wt. %
30		10 wt. %
35		5 wt. %
40		5 wt. %
45		6 wt. %
50		4 wt. %
55		5 wt. %
		3 wt. %
		3 wt. %
		10 wt. %
		4 wt. %
		3 wt. %
		6 wt. %
		3 wt. %
		3 wt. %


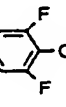


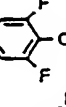

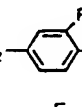
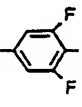

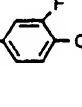

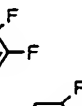




















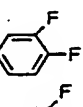


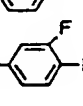
















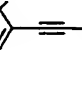


Composition example 23

5		10 wt. %
10		9 wt. %
15		5 wt. %
		5 wt. %
20		15 wt. %
		5 wt. %
25		5 wt. %
		5 wt. %
30		10 wt. %
		8 wt. %
35		3 wt. %
		4 wt. %
40		4 wt. %
		4 wt. %
45		5 wt. %
		3 wt. %

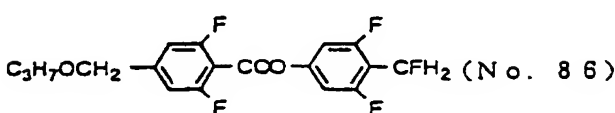
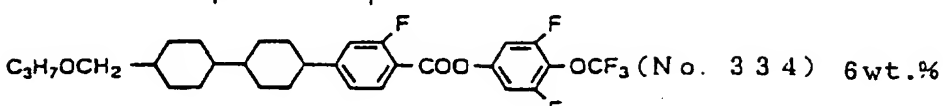
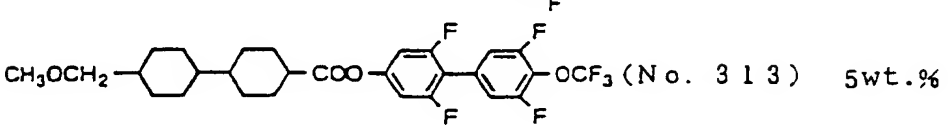





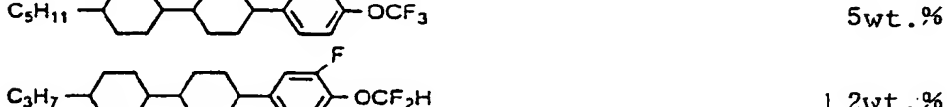
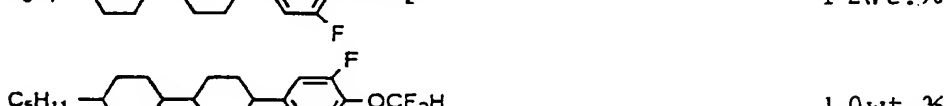




Composition example 24

5	$C_3H_7OCH_2$ —  —COO—  —OCF ₃ (N o. 18)	5wt. %
10	$C_3H_7OCH_2$ —  —  —COO—  —CF ₃ (N o. 221)	5wt. %
15	$C_5H_{11}OCH_2$ —  —  —COO—  —CCF ₂ H (N o. 251)	5wt. %
	C_3H_7 —  —  —Cl	4wt. %
20	C_3H_{11} —  —  —Cl	4wt. %
	C_2H_5 —  —  —  —Cl	5wt. %
25	C_3H_7 —  —  —  —Cl	6wt. %
	C_5H_{11} —  —  —  —Cl	5wt. %
30	C_2H_5 —  —CH ₂ CH ₂ —  — 	4wt. %
	C_2H_5 —  —  — 	6wt. %
35	C_3H_7 —  —  — 	6wt. %
	C_5H_{11} —  —  — 	12wt. %
40	C_3H_7 —  —  — 	13wt. %
45	C_5H_{11} —  —  — 	13wt. %
50	C_3H_7 —  —  —  —  —C ₂ H ₅	3wt. %
	C_3H_7 —  —  —C ₄ H ₉	4wt. %






















































Composition example 25

5	$C_3H_7OCH_2$ —  —COO—  —CN (N o. 4)	3 wt. %
10	$C_5H_{11}OCH_2$ —  —COO—  —COO—  —OCF ₃ (N o. 2 8 7)	3 wt. %
15	$C_4H_9OCH_2$ —  —CH ₂ CH ₂ —  —COO—  —CFH ₂ (N o. 2 8 2)	5 wt. %
20	$C_5H_{11}OCH_2$ —  —COO—  —CF ₃ (N o. 3 3 7)	4 wt. %
25	C_7H_{15} —  — 	5 wt. %
30	C_2H_5 —  —  — 	11 wt. %
35	C_3H_7 —  —  — 	11 wt. %
40	C_5H_{11} —  —  — 	11 wt. %
45	C_2H_5 —  —  — 	5 wt. %
50	C_3H_7 —  —  — 	5 wt. %
55	C_5H_{11} —  —  — 	10 wt. %
	C_3H_7 —  —CH ₂ CH ₂ —  — 	3 wt. %
	C_5H_{11} —  —CH ₂ CH ₂ —  — 	3 wt. %
	C_3H_7 —  —  — 	4 wt. %
	C_3H_7 —  —  —COO—  — 	4 wt. %
	C_5H_{11} —  —  —COO—  — 	3 wt. %
	C_3H_7 —  —  —OC ₂ H ₅	3 wt. %
	CH ₃ OCH ₂ —  —  —C ₃ H ₇	2 wt. %
	C_3H_7 —  —  —  —  —C ₂ H ₅	5 wt. %

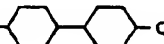
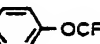

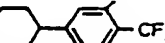


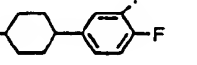
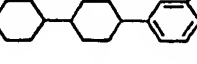
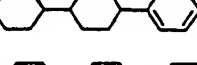
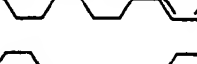
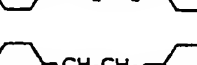
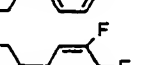
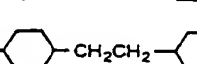
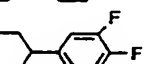
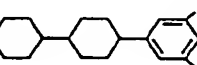

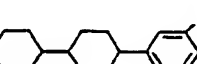



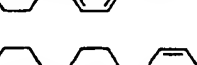

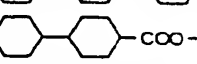
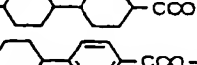
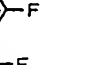
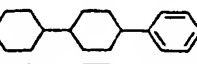





Composition example 26

5		10 wt. %
10		6 wt. %
15		5 wt. %
		12 wt. %
20		12 wt. %
		5 wt. %
25		5 wt. %
		5 wt. %
30		12 wt. %
35		10 wt. %
		5 wt. %
40		5 wt. %
45		4 wt. %
		4 wt. %

Composition example 27

5	$C_3H_7OCH_2$ —  —COO—  —OCF ₂ H (N o. 3 7 3)	8 wt. %
	$C_5H_{11}OCH_2$ —  —COO—  —OCF ₃ (N o. 3 7 4)	6 wt. %
10	$C_5H_{11}OCH_2$ —  —  —COO—  —CF ₂ H (N o. 3 8 8)	5 wt. %
	$C_3H_7OCH_2$ —  —  —COO—  —  —OCF ₃ (N o. 3 9 3)	2 wt. %
15	$C_5H_{11}OCH_2$ —  —  —  —COO—  —CFH ₂ (N o. 4 0 9)	2 wt. %
	C_5H_{11} —  — 	10 wt. %
20	C_8H_{13} —  — 	8 wt. %
	C_7H_{15} —  — 	8 wt. %
25	C_2H_5 —  —  — 	5 wt. %
	C_3H_7 —  —  — 	5 wt. %
30	C_3H_7 —  —CH ₂ CH ₂ —  — 	5 wt. %
	C_5H_{11} —  —CH ₂ CH ₂ —  — 	5 wt. %
35	C_3H_7 —  —  —CH ₂ CH ₂ — 	4 wt. %
	C_5H_{11} —  —  —CH ₂ CH ₂ — 	4 wt. %
40	C_3H_7 —  —  — 	10 wt. %
	C_5H_{11} —  —  — 	8 wt. %
45	C_3H_7 —  —  —  —  —C ₃ H ₇	3 wt. %
	C_5H_{11} —  —  —  —  —C ₃ H ₇	2 wt. %

Composition example 28

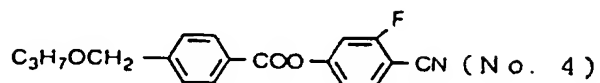
5	CH_3OCH_2 —  — COO —  — OCF_3 (N o. 3 9 1)	8 wt. %
	$\text{C}_3\text{H}_7\text{OCH}_2$ —  — COO —  — CF_2H (N o. 3 8 3)	6 wt. %
10	$\text{C}_3\text{H}_7\text{OCH}_2$ —  — COO —  — CFH_2 (N o. 3 7 8)	6 wt. %
	C_7H_{15} —  — F	7 wt. %
15	C_2H_5 —  — F	8 wt. %
	C_3H_7 —  — F	8 wt. %
20	C_5H_{11} —  — F	8 wt. %
	C_2H_5 —  — CH_2CH_2 —  — F	6 wt. %
25	C_3H_7 —  — CH_2CH_2 —  — F	3 wt. %
	C_5H_{11} —  — CH_2CH_2 —  — F	6 wt. %
30	C_2H_5 —  — F	3 wt. %
	C_3H_7 —  — F	3 wt. %
35	C_3H_7 —  — COO —  — F	4 wt. %
40	C_3H_7 —  — COO —  — F	3 wt. %
	C_3H_7 —  — Cl	5 wt. %
45	C_3H_7 —  — COO —  — F	3 wt. %
	C_5H_{11} —  — COO —  — F	3 wt. %
	C_3H_7 —  — COO —  — F	3 wt. %
50	C_3H_7 —  — CH_3	3 wt. %
	C_3H_7 —  — OCH_3	4 wt. %

55

Composition example 29

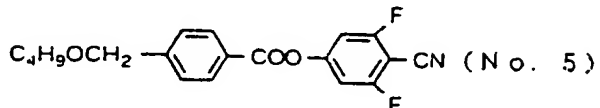
5

10



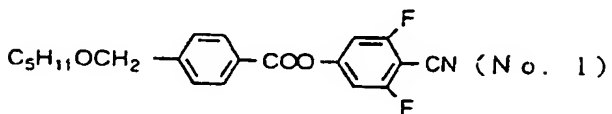
5 wt. %

15

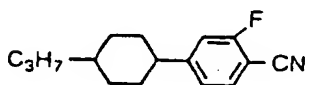


10 wt. %

20

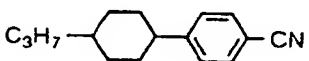


5 wt. %

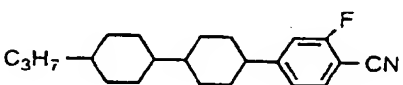


5 wt. %

25

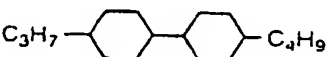


8 wt. %



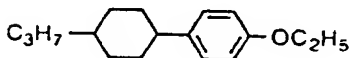
7 wt. %

30

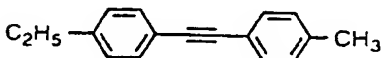


10 wt. %

35

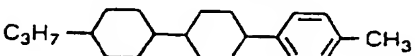


5 wt. %

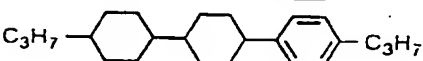


10 wt. %

40

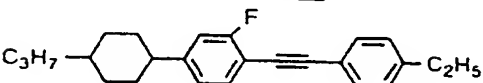


10 wt. %

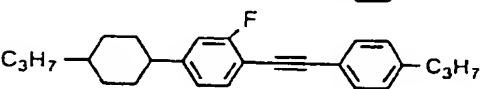


10 wt. %

45



8 wt. %

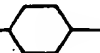

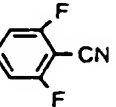


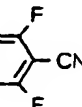

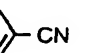

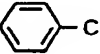

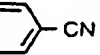

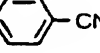

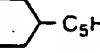











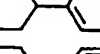


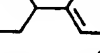








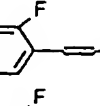
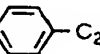

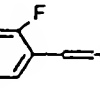
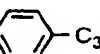


7 wt. %

50

55

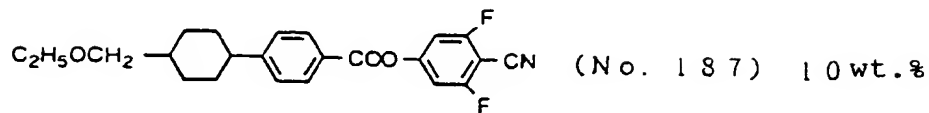
Composition example 30

5		
10	CH_3OCH_2 —  —  —COO—  —CN (No. 218) 10 wt. %	
15	$\text{C}_3\text{H}_7\text{OCH}_2$ —  —COO—  —  —CN (No. 108) 5 wt. %	
20	(E) $\text{CH}_2=\text{CHCH}_2$ —  —  —CN 11 wt. %	
25	(E) $\text{CH}_3\text{CH}=\text{CHC}_2\text{H}_5$ —  —  —CN 11 wt. %	
30	C_3H_7 —  —  —CN 11 wt. %	
35	C_5H_{11} —  —  —CN 5 wt. %	
40	C_3H_7 —  —  — C_5H_{11} 7 wt. %	
45	C_2H_5 —  — \equiv —  — OCH_3 2 wt. %	
50	C_3H_7 —  — \equiv —  — OCH_3 2 wt. %	
55	C_4H_9 —  — \equiv —  — OCH_3 2 wt. %	
	C_4H_9 —  — \equiv —  — OC_2H_5 2 wt. %	
	C_5H_{11} —  — \equiv —  — OCH_3 2 wt. %	
	C_3H_7 —  —  —  — CH_3 5 wt. %	
	C_3H_7 —  —  —  — C_3H_7 10 wt. %	
	C_3H_7 —  — CH_2CH_2 —  — \equiv —  — C_2H_5 3 wt. %	
	C_3H_7 —  — CH_2CH_2 —  — \equiv —  — C_3H_7 3 wt. %	
	C_3H_7 —  —  — \equiv —  — C_2H_5 4 wt. %	
	C_3H_7 —  —  — \equiv —  — C_3H_7 5 wt. %	

Composition example 31

5

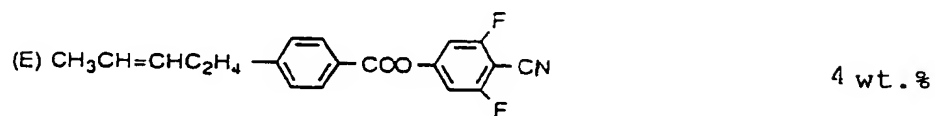
10



15



20



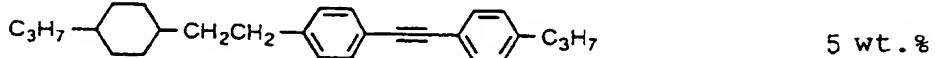
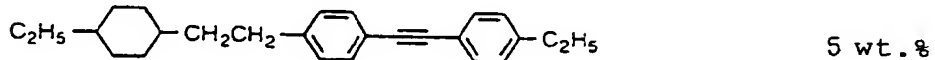
25



30



35



40



45

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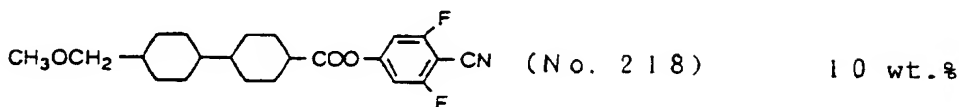
Composition example 32

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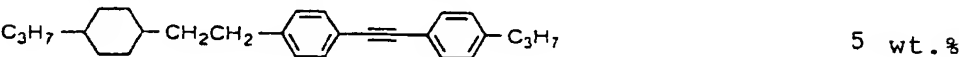
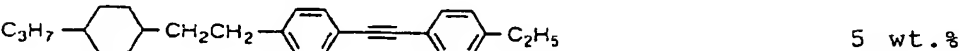
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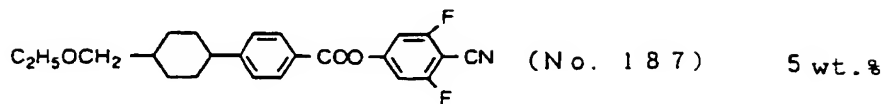
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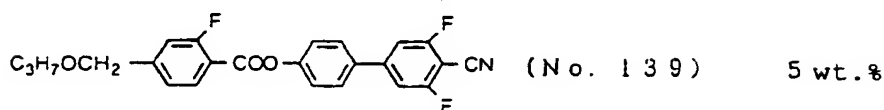
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Composition example 33

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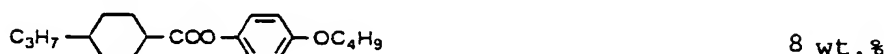
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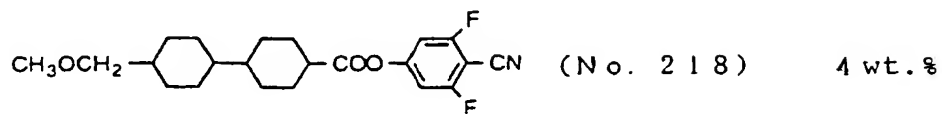


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Composition example 34

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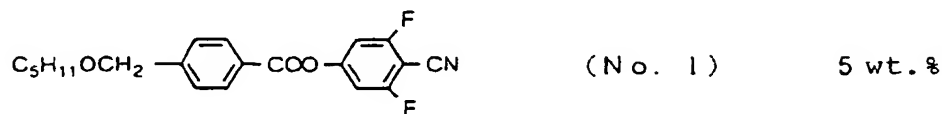
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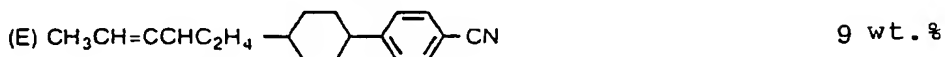
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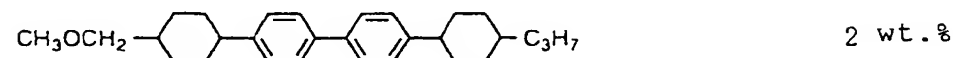
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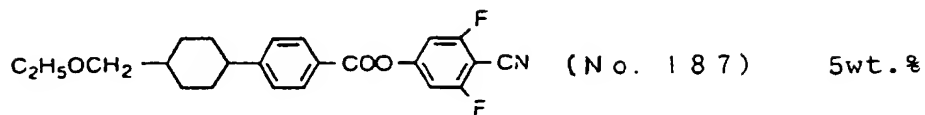


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Composition example 35

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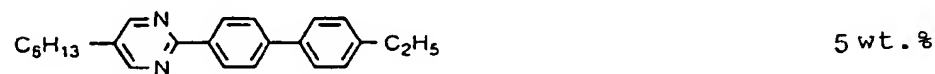
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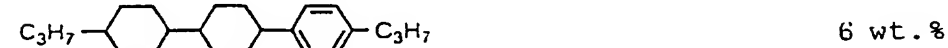
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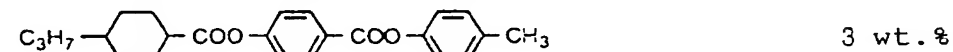
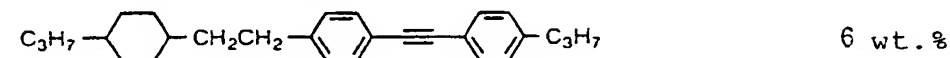
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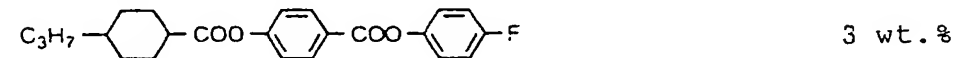
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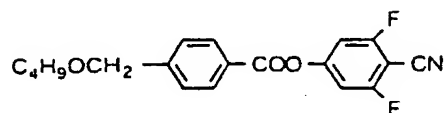
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Composition example 36

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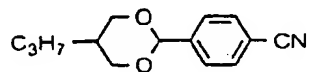
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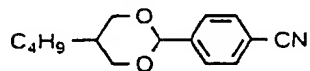
(No. 5)

6 wt. %

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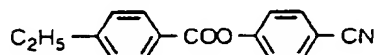


10 wt. %

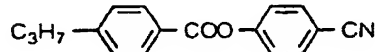


10 wt. %

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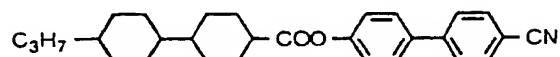


12 wt. %

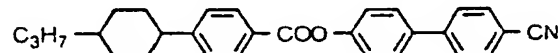


4 wt. %

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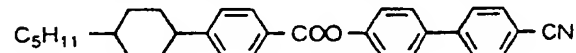


3 wt. %

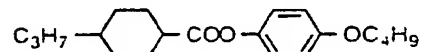


3 wt. %

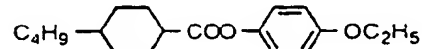
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3 wt. %

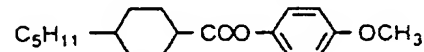


11 wt. %



8.3 wt. %

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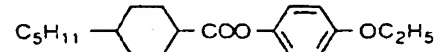


8.3 wt. %

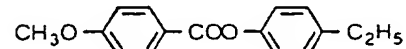


6.9 wt. %

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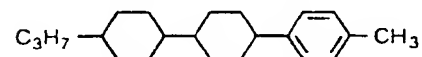


5.5 wt. %



3 wt. %

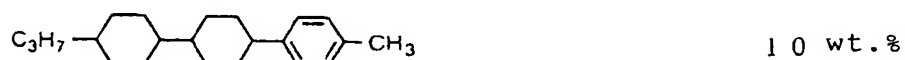
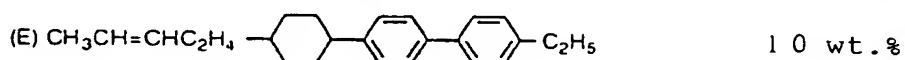
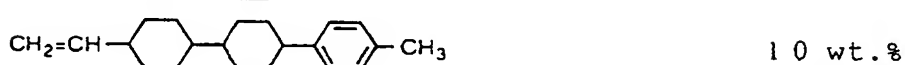
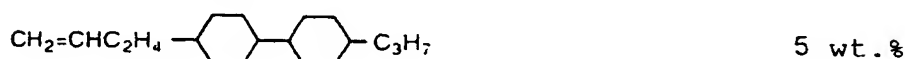
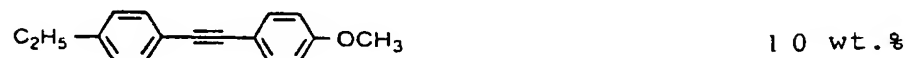
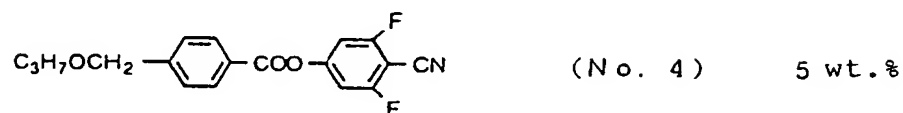
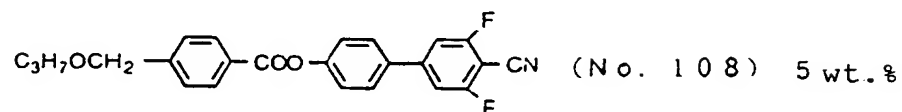
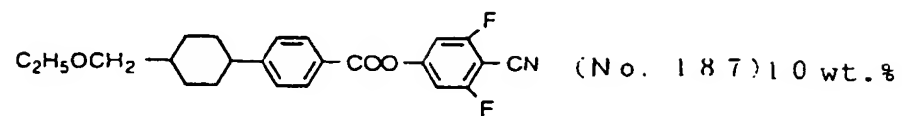
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6 wt. %

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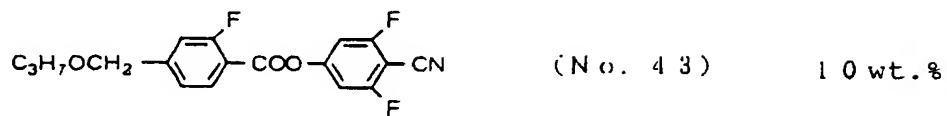
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Composition example 37

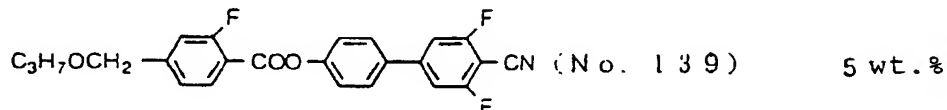
Composition example 38

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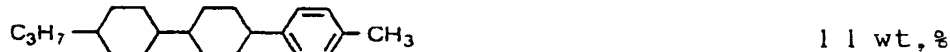
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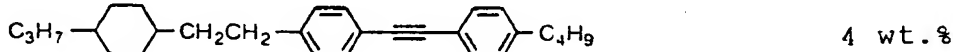
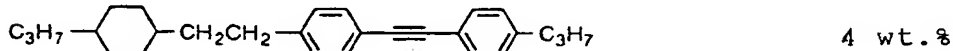
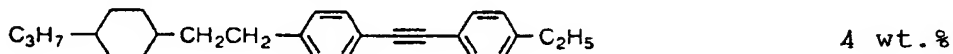
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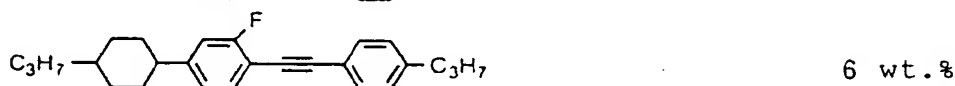
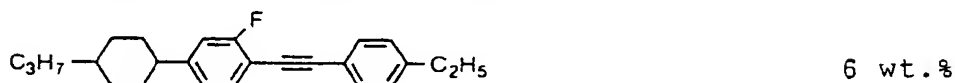
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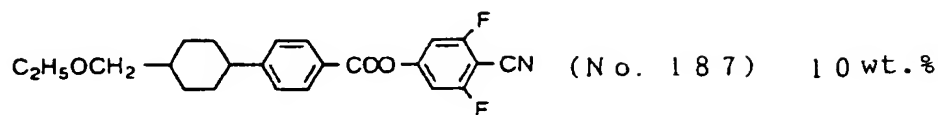
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Composition example 39

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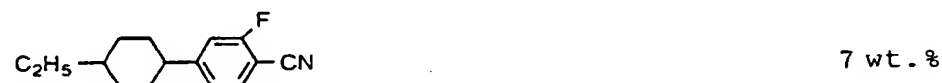
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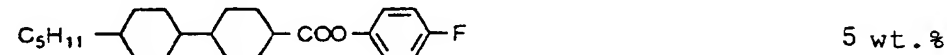
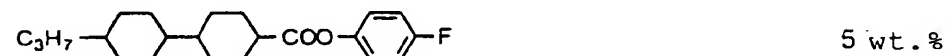
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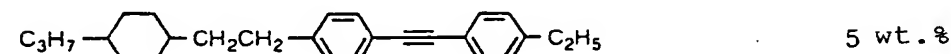
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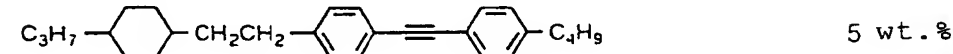
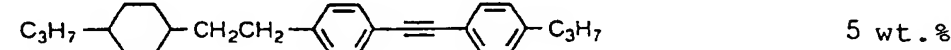
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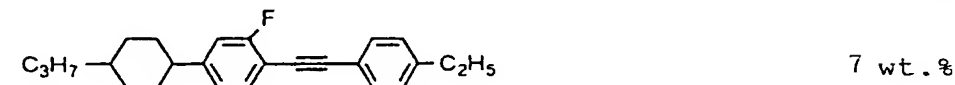
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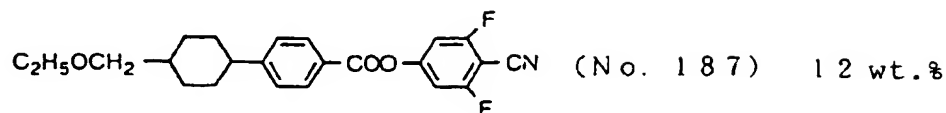
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Composition example 40

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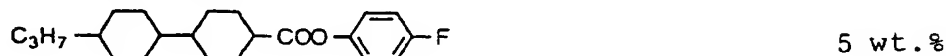
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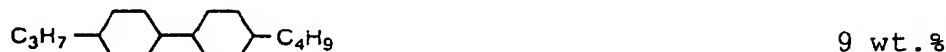
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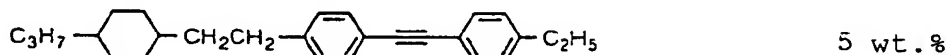
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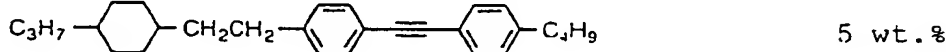
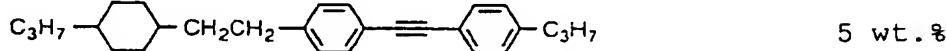
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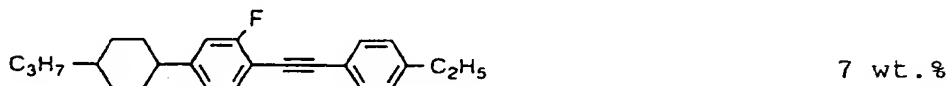
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Example

The present invention will be described in more details by way of Examples. In addition, in the respective Examples, Cr, S_A, S_B, S_C, S_X, N and Iso, respectively represent crystal, smectic A phase, smectic B phase, smectic C phase, smectic phase whose phase structure has not yet been analyzed, nematic phase, and isotropic liquid. The unit of the phase transition temperatures all refer to °C.

Example 1

Preparation of 3,5-difluoro-4-cyanophenyl 4-(pentyloxymethyl)benzoate (in the formula (1), $R=C_5H_{11}$, $\ell=1$, $m=1$, n and $o=both$, 0; $A_1=1,4$ -phenylene group, $Z_1=-COO-$, $X=CN$, and Y_1 and $Y_2 = both$, F) (Compound No. 1))

4-(Pentyloxymethyl)benzoate (1.6 g, 7.1 mmol) was mixed with thionyl chloride (1.3 g, 10.6 mmol), pyridine (0.1 mL) and toluene (3 mL), followed by reacting them at 80°C for 2 hours, and distilling off superfluous thionyl chloride and toluene under reduced pressure, to obtain raw 4-(pentyloxymethyl)benzoyl chloride.

Next, 2,6-difluoro-4-hydroxybenzonitrile (1.1 g, 7.1 mmol), pyridine (0.8 mL) and toluene (2 mL) were mixed. To this mixture was dropwise added a toluene 3 mL solution of the above 4-(pentyloxymethyl)benzoyl chloride, at room temperature for 10 minutes, followed by reacting them at 50°C for 2 hours, adding water (10 mL) to the reaction product, extracting with toluene (50 mL), washing the resulting organic layer three times with 6N-HCl, three times with 2N-NaOH and three times with water, drying over anhydrous magnesium sulfate, distilling off the solvent under reduced pressure, subjecting the residue to silica gel column chromatography (eluent: toluene), to obtain raw 3,5-difluoro-4-cyanophenyl 4-(pentyloxymethyl)benzoate (2.5 g), and recrystallizing this product from heptane/ether (6/4), to obtain the captioned compound (1.8 g) (yield: 69.0%).

Cr 32.6 ~ 33.3 Iso

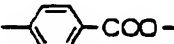
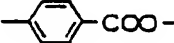
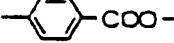
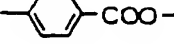
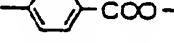
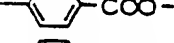
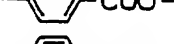

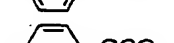

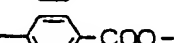
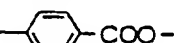
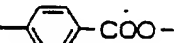
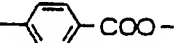
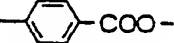
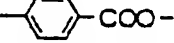
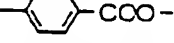
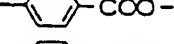



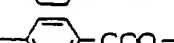


Further, the mass spector data well supported its structure.

Mass analysis: 360 (M+1).




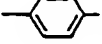
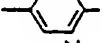
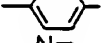

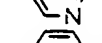
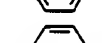
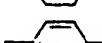

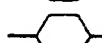
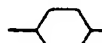
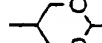
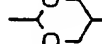
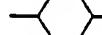
According to the process of Example 1, the following compounds (No. 2 to No. 42) are prepared.

In addition, the respective compounds were represented by extracting R, ℓ , $A=-(A_1-Z_1)_m-(A_2-Z_2)_n-(A_3-Z_3)_o$, X, Y_1 and Y_2 as parameters in the compounds expressed by the formula (1).

(This applies to the succeeding Examples.)

	No.	R	l	A	X	Y ₁	Y ₂	
5	2	CH ₃	1		CN	F	F	
	3	C ₂ H ₅	1		CN	F	F	
10	4	C ₃ H ₇	1		CN	F	F	Cr room temp. or lower Iso
	5	C ₄ H ₉	1		CN	F	F	Cr room temp. or lower Iso
	6	CH ₃	1		CF ₃	F	F	
15	7	C ₂ H ₅	1		CF ₃	F	F	
	8	C ₃ H ₇	1		CF ₃	F	F	Cr room temp. or lower Iso
20	9	C ₄ H ₉	1		CF ₃	F	F	
	10	C ₅ H ₁₁	1		CF ₃	F	F	
	11	CH ₃	1		CF ₂ H	F	F	
25	12	C ₂ H ₅	1		CF ₂ H	F	F	
	13	C ₃ H ₇	1		CF ₂ H	F	F	
30	14	C ₄ H ₉	1		CF ₂ H	F	F	
	15	C ₅ H ₁₁	1		CF ₂ H	F	F	
35	16	CH ₃	1		OCF ₃	F	F	
	17	C ₂ H ₅	1		OCF ₃	F	F	Cr room temp. or lower Iso
	18	C ₃ H ₇	1		OCF ₃	F	F	
40	19	C ₄ H ₉	1		OCF ₃	F	F	
	20	C ₅ H ₁₁	1		OCF ₃	F	F	
45	21	CH ₃	1		OCF ₂ H	F	F	
	22	C ₂ H ₅	1		OCF ₂ H	F	F	
	23	C ₃ H ₇	1		OCF ₂ H	F	F	
50	24	C ₄ H ₉	1		OCF ₂ H	F	F	
	25	C ₅ H ₁₁	1		OCF ₂ H	F	F	

55

	No.	R	l	A	X	Y ₁	Y ₂
5	26	CH ₃	1	 -COO-	CFH ₂	F	F
	27	C ₂ H ₅	1	 -COO-	CFH ₂	F	F
	28	C ₃ H ₇	1	 -COO-	CFH ₂	F	F
10	29	C ₄ H ₉	1	 -COO-	CFH ₂	F	F
	30	C ₅ H ₁₁	1	 -COO-	CFH ₂	F	F
15	31	C ₃ H ₇	1	 -COO-	CN	F	F
	32	C ₄ H ₉	1	 -COO-	CF ₃	F	F
	33	C ₅ H ₁₁	1	 -COO-	OCF ₃	F	F
20	34	C ₁₀ H ₂₁	1	 -COO-	CN	F	F
	35	C ₇ H ₁₅	1	 -COO-	CF ₃	F	F
25	36	C ₆ H ₁₃	1	 -COO-	OCF ₃	F	F
	37	C ₃ H ₇	1	 -COO-	CN	F	F
	38	C ₄ H ₉	1	 -COO-	CF ₃	F	F
30	39	C ₅ H ₁₁	1	 -COO-	OCF ₃	F	F
	40	C ₂ H ₅	1	 -COO-	CN	F	F
35	41	C ₇ H ₁₅	1	 -COO-	CF ₂ H	F	F
	42	C ₈ H ₁₇	1	-COO-	OCF ₂ H	F	F

Example 2

- 45 Preparation of 3,5-difluoro-4-cyanophenyl 2-fluoro-4-(propoxymethyl)benzoate (in the formula (1), R=C₃H₇, $\ell=1$, m=1, n and o=both, 0, A₁=2-fluoro-1,4-phenylene group, Z₁=-COO-, X=CN and Y₁ and Y₂ = both, F) (Compound No. 43)

2-Fluoro-4-(propoxymethyl)benzoate (1.5 g, 7.1 mmol) was mixed with thionyl chloride (1.3 g, 10.6 mmol) pyridine (0.1 ml) and toluene (3 ml), followed by reacting them at 80°C for 2 hours, and distilling off superfluous thionyl chloride and toluene under reduced pressure, to obtain raw 2-fluoro-4-(propoxymethyl)benzoyl chloride.

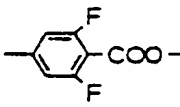
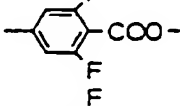
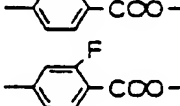
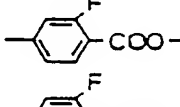
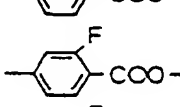
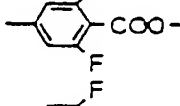
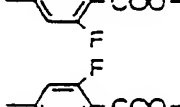
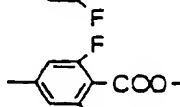
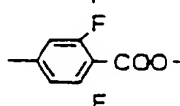
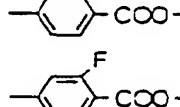
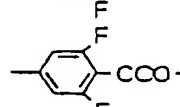




Next, 2,6-difluoro-4-hydroxybenzonitrile (1.1 g, 7.1 mmol), pyridine (0.8 ml) and toluene (2 ml) were mixed. To this mixture was dropwise added a toluene 3 ml solution of the above 2-fluoro-4-(propoxymethyl)benzoyl chloride at room temperature for 10 minutes, followed by reacting them at 50°C for 2 hours, adding water (10 ml) to the reaction product, extracting with toluene (50 ml), washing the resulting organic layer three times with 6N-HCl, three times with 2N-NaOH and three times with water, drying over anhydrous magnesium sulfate, distilling off the solvent under reduced pressure, subjecting the residue to silica gel column chromatography (eluent: toluene), to obtain raw 3,5-difluoro-4-cyanophenyl 2-fluoro-4-(propoxymethyl)-benzoate (2.4 g) and recrystallizing this product from heptane/ether (8/2), to obtain the captioned compound (1.6 g, yield: 64.8%). Cr 23.5-24.3 Iso

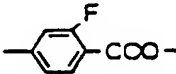
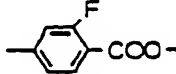
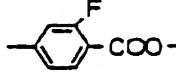
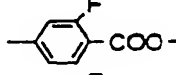
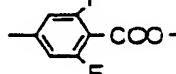
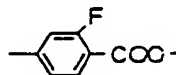
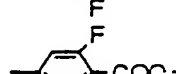
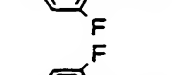
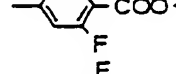
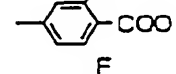
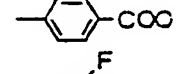
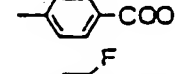
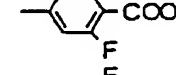
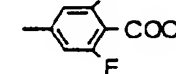
Further, the mass spector data well supported its structure.

Mass analysis: 350 (M+1)

According to the process of Example 2, the following compounds (No. 44 to No. 88) are prepared.

	No.	R	l	A	X	Y ₁	Y ₂
5	44	C ₂ H ₅	1		CN	F	F
10	45	C ₄ H ₉	1		CN	F	F
	46	C ₅ H ₁₁	1		CN	F	F
15	47	CH ₃	5		CN	F	F
	48	C ₄ H ₉	2		CN	F	F
20	49	C ₃ H ₇	1		CN	F	F
25	50	C ₄ H ₉	1		CN	F	F
	51	CH ₃	4		CN	F	F
30	52	C ₅ H ₁₁	5		CN	F	F
35	53	CH ₃	1		CF ₃	F	F
	54	C ₃ H ₇	1		CF ₃	F	F
40	55	C ₄ H ₉	1		CF ₃	F	F
	56	C ₂ H ₅	2		CF ₃	F	F
45	57	C ₃ H ₇	3		CF ₃	F	F
	58	C ₃ H ₇	1		CF ₃	F	F
50	59	C ₅ H ₁₁	1		CF ₃	F	F

No.	R	l	A	X	Y ₁	Y ₂
5 60	C ₂ H ₅	4		CF ₃	F	F
10 61	C ₃ H ₇	2		CF ₃	F	F
15 62	C ₂ H ₅	1		OCF ₃	F	F
20 63	C ₄ H ₉	1		OCF ₃	F	F
25 64	C ₅ H ₁₁	1		OCF ₃	F	F
30 65	C ₂ H ₅	2		OCF ₃	F	F
35 66	C ₃ H ₇	3		OCF ₃	F	F
40 67	C ₂ H ₅	1		OCF ₃	F	F
45 68	C ₃ H ₇	1		OCF ₃	F	F
50 69	CH ₃	2		OCF ₃	F	F
55 70	C ₄ H ₉	4		OCF ₃	F	F
71	C ₃ H ₇	1		OCF ₂ H	F	F
72	C ₄ H ₉	2		OCF ₂ H	F	F
73	C ₃ H ₇	1		OCF ₂ H	F	F
74	C ₅ H ₁₁	5		OCF ₂ H	F	F

	No.	R	l	A	X	Y ₁	Y ₂
5	75	C ₃ H ₇	1		CF ₂ H	F	F
	76	C ₅ H ₁₁	1		CF ₂ H	F	F
10	77	C ₉ H ₁₉	1		CF ₂ H	F	F
	78	C ₅ H ₁₁	2		CF ₂ H	F	F
15	79	C ₂ H ₅	1		CF ₂ H	F	F
	80	C ₃ H ₇	1		CF ₂ H	F	F
20	81	CH ₃	2		CF ₂ H	F	F
25	82	C ₃ H ₇	3		CF ₂ H	F	F
	83	C ₃ H ₇	1		CFH ₂	F	F
30	84	C ₅ H ₁₁	1		CFH ₂	F	F
35	85	C ₂ H ₅	9		CFH ₂	F	F
	86	C ₃ H ₇	1		CFH ₂	F	F
40	87	CH ₃	2		CFH ₂	F	F
45	88	C ₃ H ₇	3		CFH ₂	F	F

Example 3

Preparation of 3,5-difluoro-4-cyanophenyl 4-(ethoxyethyl)benzoate (in the formula (1), $R=C_2H_5$, $\ell=2$, $m=1$, n and $o=both$, 0, $A_1=1,4$ -phenylene group, $Z_1=-COO-$, $X=CN$, and Y_1 and $Y_2=both$, F) (Compound No. 89)

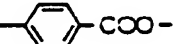
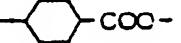
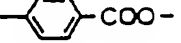
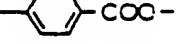
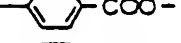
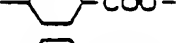




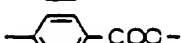
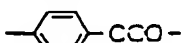
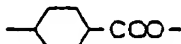
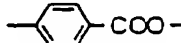
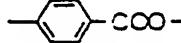
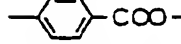
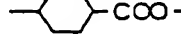

4-(Ethoxyethyl)benzoate (1.4 g, 7.2 mmol) was mixed with thionyl chloride (1.3 g, 10.8 mmol), pyridine (0.1 mL) and toluene (4 mL), followed by reacting them at 80°C for 2 hours, distilling superfluous thionyl chloride and toluene under reduced pressure, to obtain raw 4-(ethoxyethyl)benzoyl chloride.

Next, 2,6-difluoro-4-hydroxybenzonitrile (1.1 g, 7.2 mmol), pyridine (0.8 mL) and toluene (2 mL) were mixed. To this mixture was dropwise added a toluene 3 mL solution of the above 4-(ethoxyethyl)benzoyl chloride, at room temperature for 5 minutes, followed by reacting them at 50°C for 3 hours, adding water (15 mL) to the reaction product, extracting with toluene (50 mL), washing the resulting organic layer three times with 6N-HCl, three times with 2N-NaOH and three times with water, drying over anhydrous magnesium sulfate, distilling off the solvent under reduced pressure, subjecting the resulting residue to silica gel column chromatography (eluent: toluene), to obtain raw 3,5-difluoro-4-cyanophenyl 4-(ethoxyethyl)benzoate (2.2 g), and recrystallizing this product from heptane/ether (8/2), to obtain the captioned compound (1.5 g, yield: 62.8%).

Further, the mass spector data well supported its structure.

Mass analysis: 332 (M+1)

According to the process of Example 3, the following compounds (No. 90 to No. 107) are prepared.

No.	R	l	A	X	Y ₁	Y ₂
90	CH ₃	5		CN	F	F
91	C ₂ H ₅	3		CN	F	F
92	C ₄ H ₉	3		CN	F	F
93	CH ₃	9		CF ₃	F	F
94	C ₃ H ₇	2		CF ₃	F	F
95	C ₅ H ₁₁	5		CF ₃	F	F
96	C ₂ H ₅	2		CF ₂ H	F	F
97	C ₃ H ₇	3		CF ₂ H	F	F
98	C ₄ H ₉	5		CF ₂ H	F	F
99	CH ₃	2		CFH ₂	F	F
100	CH ₃	3		CFH ₂	F	F
101	C ₁₀ H ₂₁	9		CFH ₂	F	F
102	CH ₃	4		OCF ₃	F	F
103	C ₃ H ₇	2		OCF ₃	F	F
104	C ₅ H ₁₁	2		OCF ₃	F	F
105	C ₂ H ₅	5		OCF ₂ H	F	F
106	C ₄ H ₉	4		OCF ₂ H	F	F
107	C ₅ H ₁₁	2		OCF ₂ H	F	F

Example 4

Preparation of 3',5'-difluoro-4'-cyanobiphenyl-4-yl 4-(propoxymethyl)benzoate (in the formula (1), R=C₃H₇, $\ell=1$, m and n=both, 1, o=0, A₁ and A₂=both, 1,4-phenylene group, Z₁=-COO-, Z₂=covalent bond, X=CN, and Y₁ and Y₂ = both, F) (Compound No. 108)

4-(Propoxymethyl)benzoate (1.0 g, 5.2 mmol), 3',5'-difluoro-4'-cyano-4-hydroxybiphenyl (1.2 g, 5.2 mmol), DAMP (0.2 g, 1.6 mmol) and dichloromethane (20 ml) were mixed, followed by dropwise adding to the mixture, a dichloromethane 6 ml solution of DCC (1.3 g, 6.2 mmol), at room temperature for 5 minutes, stirring them as it was, for 12 hours, filtering off deposited crystals, adding toluene (50 ml) to the filtrate, washing the resulting organic layer three times with 6N-HCl, three times with 2N-NaOH and three times with water, drying over anhydrous magnesium sulfate, distilling off the solvent under reduced pressure, subjecting the residue to silica gel column chromatography (eluent: toluene), to obtain raw 3',5'-difluoro-4'-cyanobiphenyl-4-yl 4-(propoxymethyl)-benzoate (2.7 g), and recrystallizing this product from heptane/ethyl acetate (7/3), to obtain the captioned compound (1.2 g, yield: 48.3%).

Cr 91.9 to 92.8 N 121.2 Iso

Further, the mass spector data well supported its structure.

Mass analysis: 408 (M+1)

According to the process of Example 4, the following compounds (No. 109 to No. 147) are prepared.

No.	R	l	A	X	Y ₁	Y ₂
109	C ₄ H ₉	1		CN	F	F
110	C ₅ H ₁₁	1		CN	F	F
111	CH ₃	2		CN	F	F
112	C ₃ H ₇	1		CN	F	F
113	C ₅ H ₁₁	1		CN	F	F
114	C ₂ H ₅	1		CF ₃	F	F
115	C ₃ H ₇	1		CF ₃	F	F Cr 91.4-92.2 S _A 102.3-103.7 Iso
116	C ₃ H ₇	3		CF ₃	F	F
117	C ₂ H ₅	1		CF ₃	F	F
118	C ₄ H ₉	1		CF ₃	F	F
119	C ₂ H ₅	1		CF ₂ H	F	F
120	C ₃ H ₇	1		CF ₂ H	F	F
121	C ₅ H ₁₁	4		CF ₂ H	F	F
122	C ₃ H ₇	1		CF ₂ H	F	F
123	CH ₃	1		CF ₂ H	F	F
124	C ₄ H ₉	1		OCF ₃	F	F
125	C ₅ H ₁₁	1		OCF ₃	F	F
126	C ₂ H ₅	4		OCF ₃	F	F
127	CH ₃	1		OCF ₃	F	F
128	C ₄ H ₉	5		OCF ₃	F	F

	No.	R	l	A	X	Y ₁	Y ₂	
5	129	C ₃ H ₇	1		OCF ₂ H	F	F	
	130	C ₄ H ₉	1		OCF ₂ H	F	F	
10	131	CH ₃	3		OCF ₂ H	F	F	
	132	C ₂ H ₅	1		OCF ₂ H	F	F	
15	133	CH ₃	1		OCF ₂ H	F	F	
	134	C ₃ H ₇	1		CFH ₂	F	F	
	135	C ₄ H ₉	1		CFH ₂	F	F	
20	136	C ₈ H ₁₇	5		CFH ₂	F	F	
	137	C ₂ H ₅	1		CFH ₂	F	F	
25	138	C ₅ H ₁₁	1		CFH ₂	F	F	
30	139	C ₃ H ₇	1		CN	F	F	Cr 85.3-85.9 N 112.6 Iso
	140	C ₄ H ₉	1		CF ₃	F	F	
35	141	CH ₃	1		CF ₂ H	F	F	
	142	C ₂ H ₅	1		OCF ₃	F	F	
40	143	C ₅ H ₁₁	1		OCF ₂ H	F	F	
	144	C ₃ H ₇	1		CN	F	F	
45	145	C ₃ H ₇	1		CF ₃	F	F	
	146	C ₅ H ₁₁	1		CF ₃	F	F	
50	147	C ₄ H ₉	1		OCF ₃	F	F	

Example 5

Preparation of trans-4-(3,5-difluoro-4-cyanophenyl)-cyclohexyl 4-(butoxymethyl)benzoate (in the formula (1), R=C₄H₉, l=1, m and n=both, 1, o=0, A₁=1,4-phenylene group, A₂=trans-1,4-cyclohexylene group, Z₁=-COO-, Z₂=covalent bond, X=CN, and Y₁ and Y₂= both, F) (Compound No. 148)



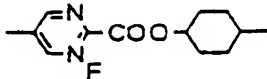


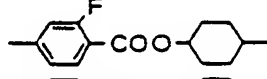

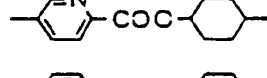
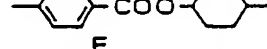
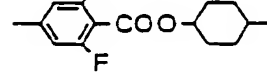

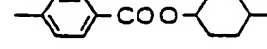
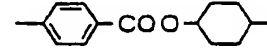
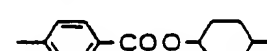
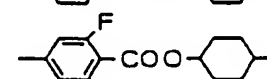
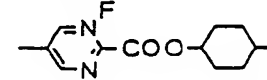
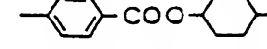
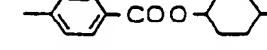
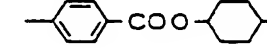
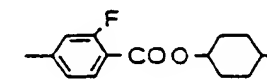
4-(butoxymethyl)benzoate (1.5 g, 7.2 mmol) was mixed with thionyl chloride (1.3 g, 10.8 mmol) pyridine (0.1 mL) and toluene (3 mL), followed by reacting them at 80°C, for 2 hours, and distilling off superfluous thionyl chloride and toluene under reduced pressure, to obtain raw 4-(butoxymethyl)benzoyl chloride.

Next, 4-(trans-4-hydroxycyclohexyl)-2,6-difluorobenzonitrile (1.7 g, 7.2 mmol), pyridine (0.8 mL) and toluene (2 mL) were mixed, followed by dropwise adding to the mixture, a toluene 3 mL solution of the above 4-(butoxymethyl)benzoyl chloride, at room temperature for 10 minutes, reacting them at 50°C for 2 hours, adding water (10 mL) to the reaction product, extracting with toluene (50 mL), washing the resulting organic layer three times with 6N-HCl, three times with 2N-NaOH and three times with water, drying over anhydrous magnesium sulfate, distilling off the solvent under reduced pressure, subjecting the residue to silica gel column chromatography (eluent: toluene), to obtain raw trans-4'-(3,5-difluoro-4-cyanophenyl)cyclohexyl 4-(butoxymethyl)benzoate (2.9 g), and recrystallizing this product from heptane/ether (6/4), to obtain the captioned compound (2.3 g, yield: 74.7%).

Further, the mass spector data well supported its structure.

Mass analysis: 428 (M+1)

According to the process of Example 5, the following compounds (No. 149 to No. 186) are prepared:

No.	R	l	A	X	Y ₁	Y ₂
5						
149	C ₂ H ₅	1		CN	F	F
150	C ₃ H ₇	1		CN	F	F
10						
151	C ₅ H ₁₁	1		CN	F	F
152	C ₂ H ₅	2		CN	F	F
15						
153	CH ₃	1		CF ₃	F	F
154	C ₃ H ₇	1		CF ₃	F	F
20						
155	C ₄ H ₉	1		CF ₃	F	F
156	C ₃ H ₁₁	9		CF ₃	F	F
25						
157	C ₂ H ₅	1		CF ₂ H	F	F
158	C ₃ H ₇	1		CF ₂ H	F	F
30						
159	C ₄ H ₉	1		CF ₂ H	F	F
160	C ₂ H ₅	3		CF ₂ H	F	F
35						
161	CH ₃	1		OCF ₃	F	F
162	C ₂ H ₅	1		OCF ₃	F	F
40						
163	C ₅ H ₁₁	1		OCF ₃	F	F
164	C ₄ H ₉	2		OCF ₃	F	F
45						
165	CH ₃	1		OCF ₂ H	F	F
166	C ₃ H ₇	1		OCF ₂ H	F	F
167	C ₅ H ₁₁	1		OCF ₂ H	F	F
50						
168	C ₁₀ H ₂₁	4		OCF ₂ H	F	F

55

	No.	R	l	A	X	Y ₁	Y ₂
5	169	C ₃ H ₇	1		CFH ₂	F	F
	170	C ₄ H ₉	1		CFH ₂	F	F
10	171	C ₅ H ₁₁	1		CFH ₂	F	F
	172	C ₆ H ₁₃	2		CFH ₂	F	F
15	173	C ₂ H ₅	1		CFH ₂	F	F
	174	C ₃ H ₇	1		CN	F	F
	175	C ₅ H ₁₁	1		CN	F	F
20	176	C ₂ H ₅	1		CF ₃	F	F
	177	C ₃ H ₇	1		CF ₃	F	F
25	178	C ₃ H ₇	1		CF ₃	F	F
	179	C ₄ H ₉	1		CF ₂ H	F	F
	180	C ₃ H ₇	3		CF ₂ H	F	F
30	181	CH ₃	1		OCF ₃	F	F
	182	C ₄ H ₉	1		OCF ₃	F	F
35	183	C ₅ H ₁₁	1		OCF ₂ H	F	F
	184	C ₈ H ₁₇	2		OCF ₂ H	F	F
	185	C ₃ H ₇	1		CFH ₂	F	F
40	186	CH ₃	7		CFH ₂	F	F

Example 6

Preparation of 3,5-difluoro-4-cyanophenyl 4-(trans-4-(ethoxymethyl)cyclohexyl)benzoate (in the formula (1), R=C₂H₅, l=1, m and n=both, 1, o=0, A₁=trans-1,4-cyclohexylene group, A₂=1,4-phenylene group, Z₁= covalent bond, Z₂=-COO-, X=CN, and Y₁ and Y₂ = both, F) (Compound No. 187)

4-(Trans-4-(ethoxymethyl)cyclohexyl)benzoate (1.9 g, 7.1 mmol) was mixed with thionyl chloride (1.3 g, 10.6 mmol), pyridine (0.1 ml) and toluene (4 ml), followed by reacting them at 80°C for 2 hours, and distilling off superfluous thionyl chloride and toluene under reduced pressure, to obtain raw 4-(trans-4-(ethoxymethyl)cyclohexyl)benzoyl chloride.

Next, 2,6-difluoro-4-hydroxybenzonitrile (1.1 g, 7.1 mmol) was mixed with pyridine (0.8 ml) and toluene (2 ml), followed by dropwise adding to the mixture, a toluene 3 ml solution of the above 4-(trans-4-(ethoxymethyl)cyclohexyl)benzoyl chloride, at room temperature for 5 minutes, reacting them at 50°C, for 3 hours, adding water (15 ml) to the reaction

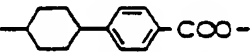
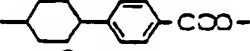
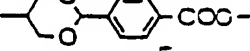
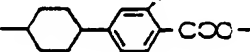
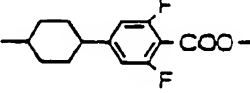
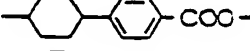
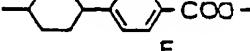
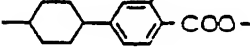
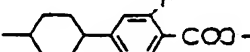
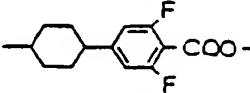
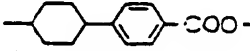
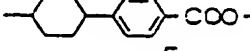
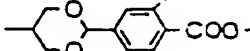

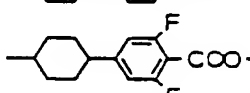
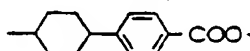
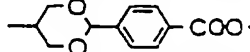
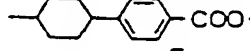
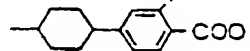
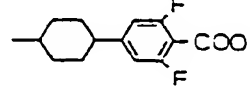
product, extracting with toluene (50 mL), washing the resulting organic layer three times with 6N-HCl, three times with 2N-NaOH and three times with water, drying over magnesium sulfate, distilling off the solvent under reduced pressure, subjecting the residue to silica gel column chromatography (eluent: toluene), to obtain raw 3,5-difluoro-4-cyanophenyl 4-(trans-4-(ethoxymethyl)cyclohexyl)benzoate (2.7 g), and recrystallizing the product from heptane/ether (7/3), to obtain the captioned compound (2.2 g, yield: 77.7%).

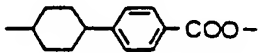
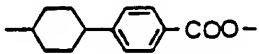
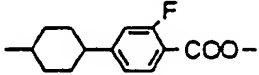
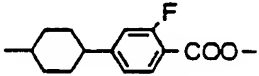
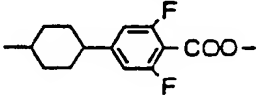
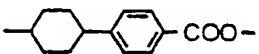
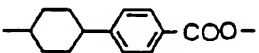
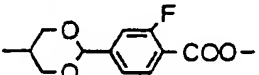
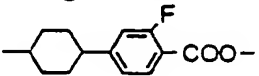
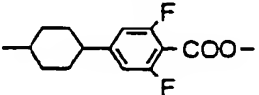
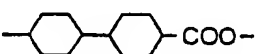
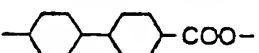
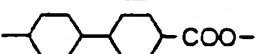
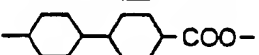
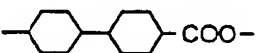
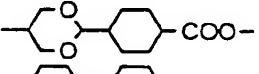
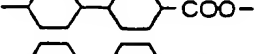
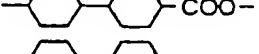
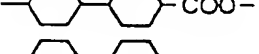
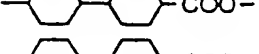

Cr 75.1 - 75.8 N 125.3 Iso

Further, the mass spector data well supported its structure.

Mass analysis: 400 (M+1)

According to the process of Example 6, the following compounds (No. 188 to No. 228) are prepared:

No.	R	l	A	X	Y ₁	Y ₂
188	C ₃ H ₇	1		CN	F	F
189	C ₄ H ₉	1		CN	F	F
190	CH ₃	9		CN	F	F
191	C ₃ H ₇	1		CN	F	F
192	C ₅ H ₁₁	1		CN	F	F
193	C ₃ H ₇	1		CF ₃	F	F
194	C ₅ H ₁₁	1		CF ₃	F	F
195	C ₃ H ₇	3		CF ₃	F	F
196	C ₂ H ₅	1		CF ₃	F	F
197	C ₄ H ₉	1		CF ₃	F	F
198	C ₂ H ₅	1		CF ₂ H	F	F
199	C ₃ H ₇	1		CF ₂ H	F	F
200	C ₅ H ₁₁	4		CF ₂ H	F	F
201	C ₃ H ₇	1		CF ₂ H	F	F
202	CH ₃	1		CF ₂ H	F	F
203	C ₂ H ₅	1		OCF ₃	F	F
204	C ₅ H ₁₁	1		OCF ₃	F	F
205	C ₆ H ₁₃	4		OCF ₃	F	F
206	C ₂ H ₅	1		OCF ₃	F	F
207	C ₄ H ₉	5		OCF ₃	F	F

	No.	R	l	A	X	Y ₁	Y ₂
5	208	C ₃ H ₇	1		OCF ₂ H	F	F
	209	C ₄ H ₉	1		OCF ₂ H	F	F
10	210	CH ₃	3		OCF ₂ H	F	F
	211	C ₂ H ₅	1		OCF ₂ H	F	F
15	212	CH ₃	1		OCF ₂ H	F	F
	213	C ₂ H ₅	1		CFH ₂	F	F
20	214	C ₅ H ₁₁	1		CFH ₂	F	F
	215	CH ₃	6		CFH ₂	F	F
25	216	C ₃ H ₇	1		CFH ₂	F	F
	217	C ₄ H ₉	1		CFH ₂	F	F
30	218	CH ₃	1		CN	F	F Cr 76.8-78.2 N 158.9 Iso
	219	C ₅ H ₁₁	1		CN	F	F
35	220	CH ₃	1		CF ₃	F	F
	221	C ₃ H ₇	1		CF ₃	F	F
40	222	C ₂ H ₅	1		CF ₂ H	F	F
	223	C ₅ H ₁₁	5		CF ₂ H	F	F
	224	C ₄ H ₉	1		OCF ₃	F	F
45	225	C ₅ H ₁₁	1		OCF ₃	F	F
	226	C ₄ H ₉	1		OCF ₂ H	F	F
	227	C ₈ H ₁₇	1		OCF ₂ H	F	F
50	228	C ₃ H ₇	1		CFH ₂	F	F

55

Example 7

Preparation of 3,5-difluoro-4-cyanophenyl 4'-(pentyloxymethyl)biphenyl-4-carboxylate (in the formula (1), $R=C_5H_{11}$, $\ell=1$, m and n =both, 1, $o=0$, A_1 and A_2 =both, 1,4-phenylene group, Z_1 =covalent bond, $Z_2=-COO-$, $X=CN$, and Y_1 and Y_2 =both, F) (Compound No. 229)

4'-(Pentyloxymethyl)biphenyl-4-carboxylate (2.1 g, 7.0 mmol) was mixed with thionyl chloride (1.3 g, 10.6 mmol), pyridine (0.1 mL) and toluene (4 mL), followed by reacting them at 80°C for 2 hours, and distilling off superfluous thionyl chloride and toluene under reduced pressure, to obtain raw 4'-(pentyloxymethyl)biphenyl-4-carbonyl chloride.

Next, 2,6-difluoro-4-hydroxybenzonitrile (1.1 g, 7.1 mmol) was mixed with pyridine (0.8 mL) and toluene (2 mL), followed by dropwise adding to the mixture, a toluene 3 mL solution of the above 4'-(pentyloxymethyl)-biphenyl-4-carbonyl chloride, at room temperature for 5 minutes, reacting them at 50°C for 3 hours, adding water (15 mL) to the reaction product, extracting with toluene (50 mL), washing the resulting organic layer, three times with 6N-HCl, three times with 2N-NaOH and three times with water, drying over anhydrous magnesium sulfate, distilling off the solvent under reduced pressure, subjecting the residue to silica gel column chromatography (eluent: toluene), to obtain raw 3,5-difluoro-4-cyanophenyl 4'-(pentyloxymethyl)biphenyl-4-carboxylate (2.9 g), and recrystallizing this product from heptane/ether (1/1), to obtain the captioned compound (2.0 g, yield: 65.3%).

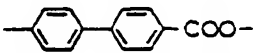
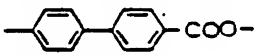
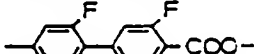

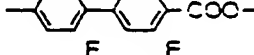
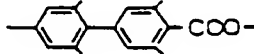


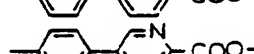
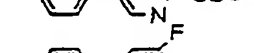
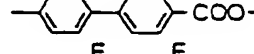
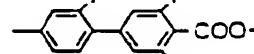
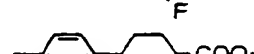
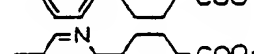



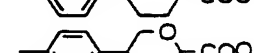

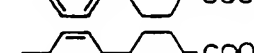

Further, the mass spector data well supported its structure.

Mass analysis: 436 ($M+1$)

According to the process of Example 7, the following compounds (No. 230 to No. 270) are prepared:

	No.	R	l	A	X	Y ₁	Y ₂
5	230	C ₃ H ₇	1		CN	F	F
	231	C ₂ H ₅	1		CN	F	F
	232	C ₃ H ₇	2		CN	F	F
10	233	C ₃ H ₇	1		CN	F	F
	234	C ₅ H ₁₁	1		CN	F	F
15	235	CH ₃	1		CF ₃	F	F
	236	C ₃ H ₇	1		CF ₃	F	F
20	237	CH ₃	3		CF ₃	F	F
	238	C ₂ H ₅	1		CF ₃	F	F
25	239	C ₄ H ₉	1		CF ₃	F	F
	240	C ₂ H ₅	1		CF ₂ H	F	F
30	241	C ₃ H ₇	1		CF ₂ H	F	F
	242	C ₅ H ₁₁	4		CF ₂ H	F	F
35	243	C ₃ H ₇	1		CF ₂ H	F	F
	244	CH ₃	1		CF ₂ H	F	F
40	245	C ₄ H ₉	1		OCF ₃	F	F
	246	C ₅ H ₁₁	1		OCF ₃	F	F
45	247	C ₂ H ₅	6		OCF ₃	F	F
	248	CH ₃	1		OCF ₃	F	F
50	249	C ₄ H ₉	5		OCF ₃	F	F

55

No.	R	l	A	X	Y ₁	Y ₂
5 250	C ₃ H ₇	1		OCF ₂ H	F	F
251	C ₃ H ₁₁	1		OCF ₂ H	F	F
10 252	CH ₃	8		OCF ₂ H	F	F
253	C ₂ H ₅	1		OCF ₂ H	F	F
15 254	CH ₃	1		OCF ₂ H	F	F
255	CH ₃	1		CFH ₂	F	F
256	C ₄ H ₉	1		CFH ₂	F	F
20 257	C ₇ H ₁₅	1		CFH ₂	F	F
258	C ₂ H ₅	1		CFH ₂	F	F
25 259	C ₃ H ₇	1		CFH ₂	F	F
30 260	C ₂ H ₅	1		CN	F	F
261	C ₄ H ₉	1		CN	F	F
262	CH ₃	4		CF ₃	F	F
35 263	C ₃ H ₇	1		CF ₃	F	F
264	C ₅ H ₁₁	1		CF ₂ H	F	F
265	C ₃ H ₇	1		CF ₂ H	F	F
40 266	C ₄ H ₉	1		OCF ₃	F	F
267	C ₉ H ₁₉	1		OCF ₃	F	F
45 268	CH ₃	1		OCF ₂ H	F	F
269	C ₄ H ₉	1		OCF ₂ H	F	F
50 270	CH ₃	1		CFH ₂	F	F

Example 8

Preparation of 3,5-difluoro-4-cyanophenyl 4-(2-(trans-4-butoxymethyl)cyclohexyl)ethyl benzoate (in the formula (1), $R=C_4H_9$, $l=1$, m and $n=both, 1$, $A_1=trans-1,4-cyclohexylene$ group, $A_2=1,4-phenylene$ group, $Z_1=-(CH_2)_2-$, $Z_2=-COO-$, $X=CN$, and Y_1 and $Y_2=both, F$) (Compound No. 271)

4-(2-Trans-4-(butoxymethyl)cyclohexyl)ethyl)benzoate (2.1 g, 7.3 mmol) was mixed with thionyl chloride (1.3 g, 10.9 mmol), pyridine (0.1 mL) and toluene (3 mL), followed by reacting them at 80°C, for 3 hours, and distilling off superfluous thionyl chloride and toluene under reduced pressure, to obtain raw 4-(2-(trans-4-(butoxymethyl)cyclohexyl)ethyl)benzoyl chloride.

Next, 2,6-difluoro-4-hydroxybenzonitrile (1.2 g, 7.4 mmol) was mixed with pyridine (0.8 mL) and toluene (2 mL), followed by dropwise adding to the mixture, a toluene 3 mL solution of the above 4-(2-(trans-4-(butoxymethyl)cyclohexyl)ethyl)benzoyl chloride at room temperature for 5 minutes, reacting them at 50°C for 2 hours, adding water (15 mL) to the reaction product, extracting with toluene (50 mL), washing the resulting organic layer three times with 6N-HCl, three times with 2N-NaOH and three times with water, drying over anhydrous magnesium sulfate, distilling off the solvent under reduced pressure, subjecting the residue to silica gel column chromatography (eluent: toluene), to obtain raw 3,5-difluoro-4-cyanophenyl 4-(2-(trans-4-(butoxymethyl)-cyclohexyl)ethyl)benzoate (2.8 g), and recrystallizing the product from heptane/ether (7/3), to obtain the captioned compound (2.5 g, yield: 80.6%).

Further, the mass spector data well supported its structure.

Mass analysis: 456 (M+1)

According to the process of Example 8, the following compounds (No. 272 to No. 293) are prepared:

No.	R	l	A	X	Y ₁	Y ₂
272	C ₅ H ₁₁	1		CN	F	F
273	C ₃ H ₇	1		CN	F	F
274	CH ₃	2		CF ₃	H	F
275	C ₃ H ₇	1		CF ₃	F	F
276	C ₂ H ₅	1		CF ₃	H	H
277	C ₃ H ₇	1		CF ₂ H	F	F
278	C ₄ H ₉	1		CF ₂ H	F	F
279	CH ₃	1		OCF ₃	F	F
280	C ₅ H ₁₁	1		OCF ₃	F	F
281	C ₃ H ₇	1		OCF ₂ H	F	F
282	C ₄ H ₉	1		CFH ₂	F	F
283	C ₂ H ₅	1		CN	F	F
284	CH ₃	1		CF ₃	F	F
285	C ₃ H ₉	1		CF ₃	H	F
286	C ₇ H ₁₅	1		CF ₂ H	F	F
287	C ₅ H ₁₁	1		OCF ₃	F	F
288	C ₄ H ₉	1		OCF ₂ H	F	F
289	CH ₃	1		CN	F	F
290	C ₃ H ₇	1		CF ₃	F	F
291	C ₅ H ₁₁	7		CFH ₂	F	F
292	C ₃ H ₇	1		OCF ₃	F	F
293	C ₂ H ₅	1		OCF ₂ H	F	F

Example 9

Preparation of 3',5'-difluoro-4'-cyanobiphenyl-4-yl 4-(trans-4-(propoxymethyl)cyclohexyl)benzoate (in the formula (1), $R=C_3H_7$, $\ell=1$, m, n and o=either, 1, A_1 =trans-1,4-cyclohexylene group, A_2 and A_3 =both, 1,4-phenylene group, Z_1 and Z_3 =both covalent bond, $Z_2=-COO-$, $X=CN$, and Y_1 and Y_2 =both, F) (Compound No. 294)

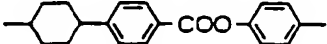
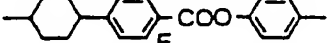
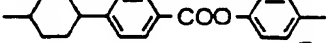
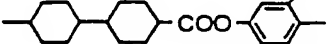
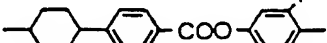
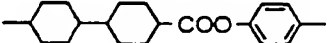
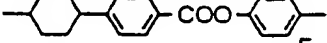
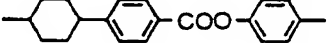
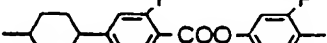
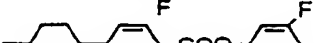
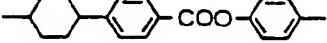
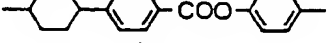
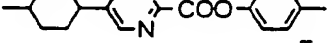
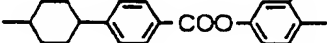
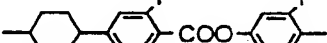
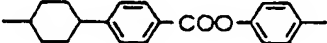
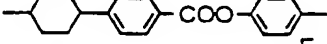
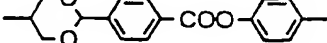
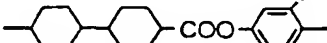
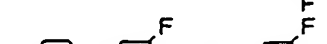
4-(Trans-4-(propoxymethyl)cyclohexyl)benzoate (2.0 g, 7.2 mmol) was mixed with thionyl chloride (1.3 g, 10.9 mmol), pyridine (0.1 mL) and toluene (4 mL), followed by reacting them at 80°C for 2 hours, and distilling off superfluous thionyl chloride and toluene under reduced pressure, to obtain raw 4-(trans-4-(propoxymethyl)cyclohexyl)benzoyl chloride.

Next, 3',5'-difluoro-4'-cyano-4-hydroxybiphenyl (1.7 g, 7.2 mmol) was mixed with pyridine (0.8 mL) and toluene (3 mL), followed by dropwise adding to the mixture, a toluene 3 mL solution of the above 4-(trans-4-(propoxymethyl)cyclohexyl)benzoyl chloride, at room temperature for 5 minutes, reacting the mixture at 50°C for 3 hours, adding water (15 mL) to the reaction product, extracting with toluene (50 mL), washing the resulting organic layer, three times with 6N-HCl, three times with 2N-NaOH and three times with water, drying over anhydrous magnesium sulfate, distilling off the solvent under reduced pressure, subjecting the residue to silica gel column chromatography (eluent: toluene), to obtain raw 3',5'-difluoro-4'-cyanobiphenyl-4-yl 4-(trans-4-(propoxymethyl)cyclohexyl)-benzoate (3.4 g), and recrystallizing this product from heptane/ether (1/1), to obtain the captioned compound (2.8 g, yield: 79.0%).

Further, the mass spector data well supported its structure.

Mass analysis: 490 (M+1)

According to the process of Example 9, the following compounds (No. 295 to No. 334) are prepared:

No.	R	l	A	X	Y ₁	Y ₂
295	C ₂ H ₅	1		CN	F	F
296	C ₅ H ₁₁	1		CN	F	F
297	CH ₃	2		CN	F	F
298	C ₃ H ₇	1		CN	F	F
299	C ₅ H ₁₁	1		CN	F	F
300	CH ₃	1		CF ₃	F	F Cr 185.3-189.3 N 224.5 Iso
301	C ₄ H ₉	1		CF ₃	F	F Cr 84.1-84.9 N 191.0 Iso
302	C ₃ H ₇	3		CF ₃	F	F
303	C ₂ H ₅	1		CF ₃	F	F
304	C ₄ H ₉	1		CF ₃	F	F
305	C ₂ H ₅	1		CF ₂ H	F	F
306	C ₃ H ₇	1		CF ₂ H	F	F
307	C ₈ H ₁₇	8		CF ₂ H	F	F
308	C ₃ H ₇	1		CF ₂ H	F	F
309	C ₂ H ₅	1		CF ₂ H	F	F
310	C ₄ H ₉	1		OCF ₃	F	F
311	C ₅ H ₁₁	1		OCF ₃	F	F
312	C ₂ H ₅	4		OCF ₃	F	F
313	CH ₃	1		OCF ₃	F	F
314	C ₄ H ₉	5		OCF ₃	F	F

No.	R	l	A	X	Y ₁	Y ₂
315	C ₃ H ₇	1		OCF ₂ H	F	F
316	C ₄ H ₉	1		OCF ₂ H	F	F
317	CH ₃	6		OCF ₂ H	F	F
318	C ₂ H ₅	1		OCF ₂ H	F	F
319	CH ₃	1		OCF ₂ H	F	F
320	C ₂ H ₅	1		CFH ₂	F	F
321	C ₅ H ₁₁	1		CFH ₂	F	F
322	C ₁₀ H ₂₁	1		CFH ₂	F	F
323	C ₃ H ₇	1		CFH ₂	F	F
324	C ₂ H ₅	1		CFH ₂	F	F
325	C ₃ H ₇	1		OCF ₃	F	F
326	C ₄ H ₉	1		OCF ₂ H	F	F
327	C ₂ H ₅	1		CN	F	F
328	C ₅ H ₁₁	1		CFH ₂	F	F
329	C ₇ H ₁₅	1		CF ₂ H	F	F
330	C ₃ H ₇	1		OCF ₃	F	F
331	CH ₃	4		OCF ₂ H	F	F
332	C ₃ H ₇	1		CN	F	F
333	C ₄ H ₉	1		CF ₃	F	F
334	C ₃ H ₇	1		OCF ₃	F	F

Example 10

Preparation of 4-(trifluoromethyl)phenyl 4-(propoxymethyl)benzoate (in the formula (1), $R=C_3H_7$, $\ell=1$, $m=1$, n and $o=both, 0$, $A_1=1,4$ -phenylene group, $Z_1=-COO-$, $X=CF_3$, and Y_1 and $Y_2=both, H$) (Compound No. 335)

4-(Propoxymethyl)benzoate (1.4 g, 7.2 mmol) was mixed with thionyl chloride (1.3 g, 10.9 mmol), pyridine (0.1 mL) and toluene (3 mL), followed by reacting them at 80°C for 2 hours, and distilling off superfluous thionyl chloride and toluene under reduced pressure, to obtain raw 4-(propoxymethyl)benzoyl chloride.

Next, 4-(trifluoromethyl)phenol (1.2 g, 7.4 mmol) was mixed with pyridine (0.8 mL) and toluene (2 mL), followed by dropwise adding to the mixture, a toluene 3 mL solution of the above 4-(propoxymethyl)benzoyl chloride at room temperature for 5 minutes, reacting them at 50°C for 2 hours, adding water (15 mL), extracting with toluene (30 mL), washing the resulting organic layer three times with 6N-HCl, three times with 2N-NaOH and three times with water, drying over anhydrous magnesium sulfate, distilling off the solvent under reduced pressure, subjecting the residue to silica gel column chromatography (eluent: toluene), to obtain raw 4-(trifluoromethyl)phenyl 4-propoxymethyl)benzoate (2.1 g), and recrystallizing this product from heptane/ether (8/2), to obtain the captioned compound (1.7 g, yield: 70.8%).

Further, the mass spector data well supported its structure.







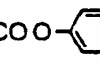

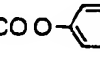

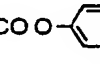

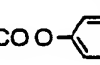

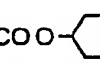

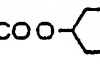

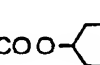
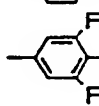
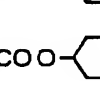

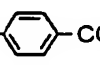

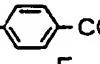

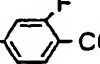

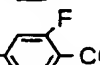

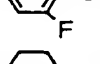

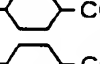

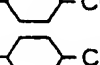
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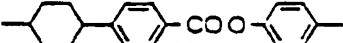
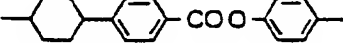
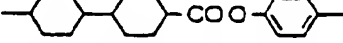
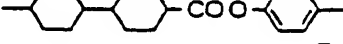
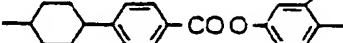
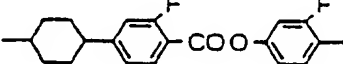
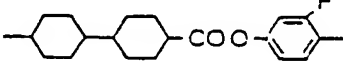
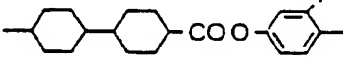
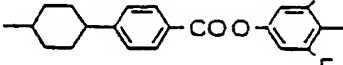
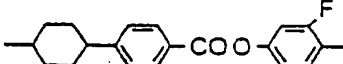
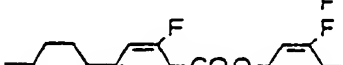
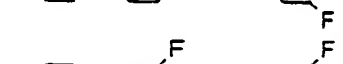
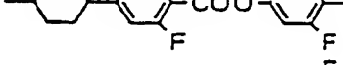
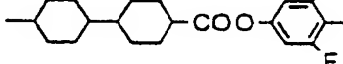
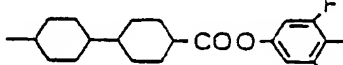
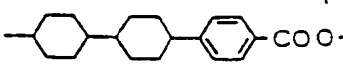
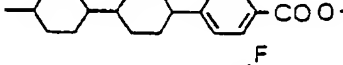
According to the process of Example 10, the following compounds (No. 336 to No. 409) are prepared:

No.	R	l	A	X	Y ₁	Y ₂
336	C ₂ H ₅	1		CF ₃	H	H
337	C ₅ H ₁₁	1		CF ₃	H	F
338	C ₃ H ₇	1		CF ₃	H	F
339	CH ₃	5		CF ₃	H	F
340	C ₄ H ₉	1		CF ₃	H	H
341	C ₃ H ₇	1		CF ₃	H	H
342	C ₄ H ₉	1		CF ₃	H	F
343	C ₉ H ₁₉	1		CF ₃	H	F
344	C ₅ H ₁₁	1		CF ₃	H	F
345	CH ₃	1		CF ₃	H	H
346	C ₃ H ₇	1		CF ₃	H	F
347	C ₄ H ₉	1		CF ₃	H	H
348	C ₂ H ₅	1		CF ₃	H	F
349	C ₄ H ₉	1		CF ₃	H	H
350	C ₅ H ₁₁	1		CF ₃	H	H
351	C ₅ H ₁₁	1		CF ₃	H	F
352	CH ₃	1		CF ₃	H	H
353	C ₂ H ₅	1		CF ₃	H	H
354	C ₃ H ₇	1		CF ₃	H	H
355	C ₅ H ₁₁	1		CF ₃	H	F

Cr 69.6-70.2 S_A 72.9-
73.1 N 75.6 Iso

	No.	R	l	A	X	Y ₁	Y ₂
5	356	C ₃ H ₇	1		CF ₃	H	H
	357	C ₂ H ₅	1		CF ₃	H	F
10	358	CH ₃	1		CF ₃	H	F
	359	C ₄ H ₉	1		CF ₃	H	H
15	360	C ₆ H ₁₃	1		CF ₃	H	F
	361	C ₅ H ₁₁	1		CF ₃	H	H
20	362	C ₃ H ₇	1		CF ₃	H	F
	363	C ₄ H ₉	1		CF ₃	H	H
25	364	C ₂ H ₅	1		CF ₃	H	F
	365	CH ₃	1		CF ₃	H	H
30	366	C ₂ H ₅	1		CF ₃	H	H
35	367	C ₃ H ₇	7		CF ₃	H	F
40	368	C ₂ H ₅	1		CF ₃	H	H
	369	C ₄ H ₉	1		CF ₃	H	H
45	370	C ₃ H ₇	1		CF ₃	H	H
	371	C ₄ H ₉	1		CF ₃	H	F
50	372	C ₅ H ₁₁	1		CF ₃	H	F

No.	R	l	A	X	Y ₁	Y ₂
373	C ₃ H ₇	1	 -COO-	OCF ₂ H	H	H
374	C ₅ H ₁₁	1	 -COO-	OCF ₃	H	H Cr 68.2-68.5 Iso
375	C ₂ H ₅	7	 -COO-	CFH ₂	H	F
376	C ₃ H ₇	1	 -COO-	OCF ₃	H	F Cr 30.5-31.8 Iso
377	C ₄ H ₉	1	 -COO-	OCF ₂ H	H	H
378	C ₃ H ₇	1	 -COO- 	CFH ₂	H	H
379	C ₄ H ₉	1	 -COO- 	CF ₂ H	H	F
380	C ₉ H ₁₉	1	 -COO- 	OCF ₂ H	H	F
381	C ₅ H ₁₁	1	 -COO- 	OCF ₃	H	F
382	CH ₃	1	 -COO- 	CFH ₂	H	H
383	C ₃ H ₇	1	 -COO- 	CF ₂ H	H	F
384	C ₄ H ₉	1	 -COO- 	CFH ₂	H	H
385	C ₂ H ₅	1	 -COO- 	OCF ₂ H	H	F
386	C ₄ H ₉	1	 -  -COO-	CFH ₂	H	H
387	C ₃ H ₇	1	 -  -COO-	OCF ₃	H	F Cr 75.1-76.0(SA 61.7) N 96.2 Iso
388	C ₅ H ₁₁	1	 -  -COO-	CF ₂ H	H	F
389	CH ₃	1	 -  -COO-	OCF ₃	H	H
390	C ₂ H ₅	1	 -  -COO-	CF ₂ H	H	H
391	CH ₃	1	 -  -COO-	OCF ₃	H	H
392	CH ₃	1	 -  -COO-	OCF ₃	H	F Cr 47.7-48.9 N 146.0 Iso

No.	R	l	A	X	Y ₁	Y ₂
393	C ₃ H ₇	1		OCF ₃	H	H
394	C ₂ H ₅	1		CF ₂ H	H	F
395	CH ₃	1		OCF ₃	H	H
396	C ₄ H ₉	1		OCF ₂ H	H	H
397	C ₇ H ₁₅	1		CFH ₂	H	F
398	C ₅ H ₁₁	1		CF ₂ H	H	H
399	C ₃ H ₇	1		OCF ₃	H	F
400	C ₄ H ₉	1		CFH ₂	H	F
401	C ₂ H ₅	1		OCF ₃	H	F
402	CH ₃	3		CF ₂ H	H	F
403	C ₂ H ₅	1		OCF ₂ H	H	H
404	C ₃ H ₇	2		OCF ₃	H	F
405	C ₂ H ₅	1		CFH ₂	H	F
406	C ₄ H ₉	1		OCF ₂ H	H	H
407	C ₃ H ₇	1		OCF ₃	H	H
408	C ₄ H ₉	1		CF ₂ H	H	F
409	C ₅ H ₁₁	1		CFH ₂	H	F

Hereinafter, examples using the compound of the present invention as a component of liquid crystal compositions will be described.

In the use examples, NI, Δε, Δn, η and V₁₀, respectively represent clearing point (°C), dielectric anisotropy value, optical anisotropy value, viscosity at 20°C (mPa·s) and threshold voltage (V).

Example 11 (Use example 1)

A liquid crystal composition consisting of the following cyanophenylcyclohexane group compounds:

4-(trans-4-propylcyclohexyl)benzonitrile	24% by weight,
4-(trans-4-pentylcyclohexyl)benzonitrile	36% by weight,
4-(trans-4-heptylcyclohexyl)benzonitrile	25% by weight,
and 4-(trans-4-pentylcyclohexyl)-4'-cyanobiphenyl	15% by weight,

has the following physical properties:

NI: 72.4, $\Delta\epsilon$: 11.0, Δn : 0.137, η : 27.0 and

a V_{10} in a cell thickness of 9 μm : 1.78.

Compound No. 4 (15% by weight) was blended with the above composition (85% by weight), to obtain a nematic liquid crystal composition (a). The values of the physical properties of this liquid crystal composition were as follows:

NI: 54.8, $\Delta\epsilon$: 16.9, Δn : 0.130, η : 31.9 and

V_{10} in a cell thickness of 8.8 μm : 1.02.

This composition was allowed to stand in a freezer at -20°C, but deposition of crystals and smectic phase were not observed even after 60 days.

Example 12 (Use example 2)

A liquid crystal composition consisting of the following cyanophenylcyclohexane group compounds:

4-(trans-4-propylcyclohexyl)benzonitrile	30% by weight,
4-(trans-4-pentylcyclohexyl)benzonitrile	40% by weight, and
4-(trans-4-heptylcyclohexyl)benzonitrile	30% by weight,

has the following physical properties:

NI: 52.3, $\Delta\epsilon$: 10.7, Δn : 0.119, η : 21.7, and

V_{10} in a cell thickness of 9 μm : 1.60.

A compound (No. 187) of Example 6 (15% by weight) was blended with the above composition (85% by weight), to prepare a nematic liquid crystal composition (b). The physical properties of this composition were as follows:

NI: 55.6, $\Delta\epsilon$: 17.5, Δn : 0.121, η : 33.1 and

V_{10} in a cell thickness of 8.8 μm : 1.06.

This composition was allowed to stand in a freezer at -20°C, but deposition of crystals and smectic phase were not observed even after 60 days.

Example 13 (Use example 3)

The physical properties of nematic liquid crystal composition of composition example 19 were as follows:

NI: 92.1, Δn : 0.143, η : 32.7, and V_{10} in a cell thickness of 8.9 μm : 1.20.

This composition was allowed to stand in a freezer at -20°C, but any deposition of crystals and smectic phase were not observed even after 60 days.

Example 14 (Use example 4)

The physical properties of nematic liquid crystal composition of composition example 29 were as follows:

NI: 79.1, $\Delta\epsilon$: 11.9, Δn : 0.139, η : 25.8, V_{th} : 1.32.

This composition was allowed to stand in a freezer at -20°C, but any deposition of crystals and smectic phase were not observed even after 60 days.

Example 15 (Use example 5)

The physical properties of nematic liquid crystal composition of composition example 30 were as follows:

NI: 97.2, $\Delta\epsilon$: 21.1, Δn : 0.155, η : 25.3, V_{th} : 1.22.

This composition was allowed to stand in a freezer at -20°C, but any deposition of crystals and smectic phase were not observed even after 60 days.

Example 16 (Use example 6)

The physical properties of nematic liquid crystal composition of composition example 31 were as follows:

NI: 98.5, $\Delta\epsilon$: 11.8, Δn : 0.139, η : 36.2, V_{th} : 1.63.

This composition was allowed to stand in a freezer at -20°C, but any deposition of crystals and smectic phase were not observed even after 60 days.

Example 17 (Use example 7)

The physical properties of nematic liquid crystal composition of composition example 32 were as follows:

NI: 81.9, $\Delta\epsilon$: 22.6, Δn : 0.147, η : 49.3, V_{th} : 1.07.

This composition was allowed to stand in a freezer at -20°C, but any deposition of crystals and smectic phase were not observed even after 60 days.

Example 18 (Use example 8)

The physical properties of nematic liquid crystal composition of composition example 33 were as follows:

NI: 81.0, $\Delta\epsilon$: 11.1, Δn : 0.126, η : 35.8, V_{th} : 1.53.

This composition was allowed to stand in a freezer at -20°C, but any deposition of crystals and smectic phase were not observed even after 60 days.

Example 19 (Use example 9)

The physical properties of nematic liquid crystal composition of composition example 34 were as follows:

NI: 76.7, $\Delta\epsilon$: 12.8, Δn : 0.123, η : 28.8, V_{th} : 1.58.

This composition was allowed to stand in a freezer at -20°C, but any deposition of crystals and smectic phase were not observed even after 60 days.

Example 20 (Use example 10)

The physical properties of nematic liquid crystal composition of composition example 35 were as follows:

NI: 85.0, $\Delta\epsilon$: 12.7, Δn : 0.141, η : 29.3, V_{th} : 1.51.

This composition was allowed to stand in a freezer at -20°C, but any deposition of crystals and smectic phase were not observed even after 60 days.

Example 21 (Use example 11)

The physical properties of nematic liquid crystal composition of composition example 36 were as follows:

NI: 74.9, $\Delta\epsilon$: 13.6, Δn : 0.131, η : 45.3, V_{th} : 1.33.

This composition was allowed to stand in a freezer at -20°C, but any deposition of crystals and smectic phase were not observed even after 60 days.

Example 22 (Use example 12)

The physical properties of nematic liquid crystal composition of composition example 37 were as follows:

NI: 76.1, $\Delta\epsilon$: 13.3, Δn : 0.135, η : 23.4, V_{th} : 1.24.

This composition was allowed to stand in a freezer at -20°C, but any deposition of crystals and smectic phase were not observed even after 60 days.

Example 23 (Use example 13)

The physical properties of nematic liquid crystal composition of composition example 38 were as follows:

NI: 92.2, $\Delta\epsilon$: 10.5, Δn : 0.163, η : 18.6, V_{th} : 1.68.

This composition was allowed to stand in a freezer at -20°C , but any deposition of crystals and smectic phase were not observed even after 60 days.

Example 24 (Use example 14)

The physical properties of nematic liquid crystal composition of composition example 39 were as follows:

NI: 89.3, $\Delta\epsilon$: 16.8, Δn : 0.141, η : 29.6, V_{th} : 1.30.

This composition was allowed to stand in a freezer at -20°C , but any deposition of crystals and smectic phase were not observed even after 60 days.

Example 25 (Use example 15)

The physical properties of nematic liquid crystal composition of composition example 40 were as follows:

NI: 92.1, $\Delta\epsilon$: 17.1, Δn : 0.143, η : 32.7, V_{th} : 1.20.

This composition was allowed to stand in a freezer at -20°C , but any deposition of crystals and smectic phase were not observed even after 60 days.

Example 26

With regard to the liquid crystal composition (a) obtained in Example 11, and Comparative liquid crystal composition (c) obtained in the same manner as in Example 11 except that 3,5-difluoro-4-cyanophenyl=4-pentyl benzoate outside the range of the present invention was used in place of No. 4 compound, the respective temperature changes in the threshold voltages were measured. The results are shown in Fig. 1.

In addition, in the figure, the longitudinal axis refers to $V_{10}(\text{V})$, and the lateral axis refers to a value ($^{\circ}\text{C}$) obtained by subtracting the clearing point (NI) of the liquid crystal composition from the measured temperature (T).

As apparent from the figure, the change in the threshold voltage within the measured temperatures, is $6.3 \text{ mV}/^{\circ}\text{C}$ in the case of the liquid crystal composition (a) of the present invention, whereas it is $7.3 \text{ mV}/^{\circ}\text{C}$ in the case of the liquid crystal composition (c); thus it was found that the temperature change of the liquid crystal composition of the present invention was smaller. Accordingly, it is possible to provide a liquid crystal display element having a higher grade.

Example 27

With regard to the liquid crystal composition (b) of the present invention obtained in Example 12, and a comparative liquid crystal composition (d) obtained in the same manner as in Example 12 except that 3,5-difluoro-4-cyanophenyl 4-(trans-4-butylcyclohexyl)benzoate outside the range of the present invention was used in place of compound No. 187, the respective temperature changes in the threshold voltages were measured. The results are shown in Fig. 2.

As apparent from the figure, the change in the threshold voltage within the measured temperatures, is $5.3 \text{ mV}/^{\circ}\text{C}$ in the case of the liquid crystal composition (b) of the present invention, whereas it is $6.3 \text{ mV}/^{\circ}\text{C}$ in the case of the liquid crystal composition (d); thus it was found that the temperature change of the liquid crystal composition of the present invention was smaller, as in Example 26. Accordingly, it is possible to provide a liquid crystal display element having a higher grade.

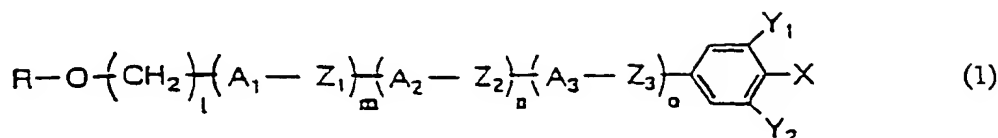
Effectiveness of the Invention

As seen from the foregoing any of the compounds of the present invention have a large dielectric anisotropy value, a small change in the threshold voltage value depending upon temperatures, and an improved solubility in other liquid crystal materials at lower temperatures.

Accordingly, when the compound of the present invention is used as a component of liquid crystal compositions, it is characterized by exhibiting a superior solubility in other liquid crystal materials, and when six-membered ring, substituent and/or bonding group as molecule-constituting elements are adequately selected, it is possible to provide novel liquid crystal compositions having desired physical properties.

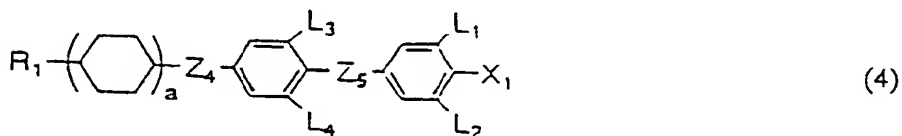
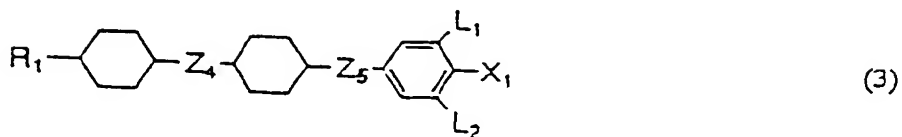
Claims

1. A liquid crystalline compound expressed by the formula (1):



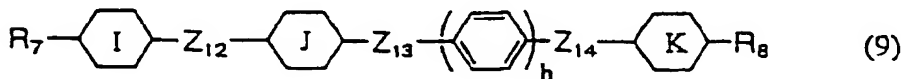
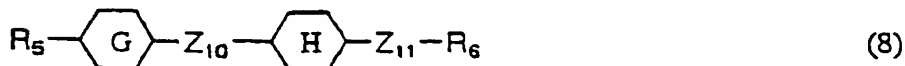
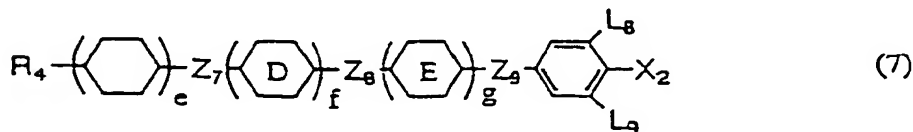
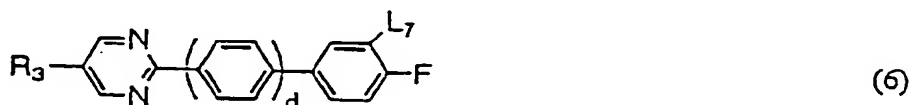
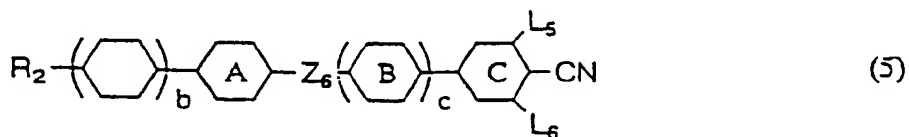
wherein R represents an alkyl group of 1 to 10 carbon atoms; l represents an integer of 1 to 9; m , n and o each independently represent 0 or 1; A_1 , A_2 and A_3 each independently represent trans-1,4-cyclohexylene group, or 1,4-phenylene group, pyrimidine-2,5-diyl group, pyridine-2,5-diyl group or 1,3-dioxane-2,5-diyl group, one or more hydrogen atoms on the rings of which groups may be replaced by F; Z_1 , Z_2 and Z_3 each independently represent $-COO-$, $-(CH_2)_2-$ or a covalent bond at least one of which represent $-COO-$; X represents CN, CF_3 , CF_2H , CFH_2 , OCF_3 or OCF_2H ; Y_1 and Y_2 each independently represent H or F, but when X represents CN, Y_1 and Y_2 both represent F.

2. A liquid crystalline compound according to claim 1, wherein m represents 1; n and o represents 0; A_1 represents 1,4-phenylene group, one or more hydrogen atoms on the ring of which group may be replaced by F; and Z_1 represents $-COO-$.
3. A liquid crystalline compound according to claim 1, wherein m and n each represent 1; o represents 0; A_1 and A_2 each represent 1,4-phenylene group, one or more hydrogen atoms on the ring of which group may be replaced by F; and Z_1 represents $-COO-$.
4. A liquid crystalline compound according to claim 1, wherein m and n each represent 1; o represents 0; A_1 represents 1,4-phenylene group, one or more hydrogen atoms on the ring of which group may be replaced by F; A_2 represents trans-1,4-cyclohexylene group; and Z_1 represents $-COO-$.
5. A liquid crystalline compound according to claim 1, wherein m and n each represent 1; o represents 0; A_1 represents trans-1,4-cyclohexylene group; A_2 represents 1,4-phenylene group, one or more hydrogen atoms on the ring of which group may be replaced by F; and Z_2 represents $-COO-$.
6. A liquid crystalline compound according to claim 1, wherein m and n each represent 1; o represents 0; A_1 and A_2 each represent 1,4-phenylene group, one or more hydrogen atoms on the ring of which group may be replaced by F; and Z_2 represents $-COO-$.
7. A liquid crystalline compound according to claim 1, wherein m and n each represent 1; o represents 0; A_1 represent trans-1,4-cyclohexylene; A_2 represents 1,4-phenylene group, one or more hydrogen atoms on the ring of which group may be replaced by F; Z_1 represents $-(CH_2)_2-$; and Z_2 represents $-COO-$.
8. A liquid crystalline compound according to claim 1, wherein m , n and o each represent 1; A_1 represents trans-1,4-cyclohexylene group; A_2 and A_3 each represent 1,4-phenylene group, one or more hydrogen atoms on the ring of which group may be replaced by F; Z_1 and Z_3 each represent a covalent bond; and Z_2 represents $-COO-$.
9. A liquid crystal composition characterized by containing at least one member of the liquid crystalline compound set forth in either one of claims 1 to 8.
10. A liquid crystal composition characterized by containing as a first component, at least one member of the liquid crystalline compounds set forth in either one of claims 1 to 8, and as a second component, at least one member selected from the group consisting of compounds expressed by the following formulas (2), (3) and (4):



In these formulas, R_1 represents an alkyl group of 1 to 10 carbon atoms; X_1 represents F, Cl, OCF_3 , OCF_2H , CF_3 , CF_2H or CFH_2 ; L_1 , L_2 , L_3 and L_4 each independently represent H or F; Z_4 and Z_5 each independently represent $-(\text{CH}_2)_2-$, $-\text{CH}=\text{CH}-$ or a covalent bond; and a represents 1 or 2.

11. A liquid crystal composition characterized by containing as a first component, at least one member of the liquid crystalline compounds set forth in either one of claims 1 to 8, and as a second component, at least one member selected from the group consisting of compounds expressed by the following formulas (5), (6), (7), (8) and (9):



In the formula (5), R_2 represents F, an alkyl group of 1 to 10 carbon atoms or an alkenyl group of 2 to 10 carbon atoms, one or more non-adjacent methylene groups ($-\text{CH}_2-$) in the alkyl group or the alkenyl group may be replaced by oxygen ($-\text{O}-$); ring A represents trans-1,4-cyclohexylene group, 1,4-phenylene group, pyrimidine-2,5-diyl group or 1,3-dioxane-2,5-diyl group; ring B represents trans-1,4-cyclohexylene group, 1,4-phenylene group or pyrimidine-

2,5-diyl group; ring C represent trans-1,4-cyclohexylene group or 1,4-phenylene group; Z_6 represents $-(CH_2)_2-$, $-COO-$ or a covalent bond; L_5 and L_6 each independently represent H or F; b and c each independently represent 0 or 1.

In the formula (6), R_3 represents an alkyl group of 1 to 10 carbon atoms; L_7 represents H or F; and d represents 0 or 1.

In the formula (7), R_4 represents an alkyl group of 1 to 10 carbon atoms; rings D and E each independently represent trans-1,4-cyclohexylene group or 1,4-phenylene group; Z_7 and Z_8 each independently represent $-COO-$ or a covalent bond; Z_9 represents $-COO-$ or $-C=C-$; L_8 and L_9 each independently represent H or F; X_2 represents F, OCF_3 , OCF_2H , CF_3 , CF_2H or CFH_2 , but when X_2 represents OCF_3 , OCF_2H_2 , CF_3 , CF_2H or CFH_2 , L_8 and L_9 both represent H; and e, f and g each independently represent 0 or 1.

In the formula (8), R_5 and R_6 each independently represent an alkyl group of 1 to 10 carbon atoms or an alkenyl group of 2 to 10 carbon atoms, and in either of the groups, one or more non-adjacent methylene groups $(-CH_2-)$ may be replaced by oxygen $(-O-)$;

ring G represents trans-1,4-cyclohexylene group, 1,4-phenylene group or pyrimidine-2,5-diyl group; ring H represents trans-1,4-cyclohexylene group or 1,4-phenylene group; Z_{10} represents $-C=C-$, $-COO-$, $-(CH_2)_2-$, $-CH=CH-C=C-$ or a covalent bond; and Z_{11} represents $-COO-$ or a covalent bond.

In the formula (9), R_7 and R_8 each independently represent an alkyl group of 1 to 10 carbon atoms or an alkenyl group of 2 to 10 carbon atoms, and in either of the groups, one or more non-adjacent methylene groups $(-CH_2-)$ may be replaced by oxygen $(-O-)$; ring I represents trans-1,4-cyclohexylene group, 1,4-phenylene group or pyrimidine-2,5-diyl group; ring J represents trans-1,4-cyclohexylene group, or 1,4-phenylene group, or pyrimidine-2,5-diyl group, one or more hydrogen atoms on the rings of which groups may be replaced by F, ring K represents trans-1,4-cyclohexylene group or 1,4-phenylene group; Z_{12} and Z_{14} each independently represent $-COO-$, $-(CH_2)_2-$ or a covalent bond; Z_{13} represents $-CH=CH-$, $-C=C-$, $-COO-$ or a covalent bond; and h represents 0 or 1.

12. A liquid crystal composition, characterized by containing as a first component, at least one member of the liquid crystalline compounds set forth in either one of claims 1 to 8, as a part of a second component, at least one member selected from the group consisting of the compounds expressed by the formulas (2), (3) and (4) set forth in claim 10, and as another part of the second component, at least one member selected from the group consisting of the compounds expressed by the formulas (5), (6), (7), (8) and (9) set forth in claim 11.

13. A liquid crystal display element composed of a liquid crystal composition set forth in either one of claim 9 to 12.

14. A phenol derivative as a raw material for a liquid crystalline compound, expressed by the formula (1-a):



wherein A_3 represents trans-1,4-cyclohexylene group or 1,4-phenylene group one or more of hydrogen atoms on the ring of which group may be replaced by F, X represents CF_3 , CF_2H , CFH_2 , OCF_3 or OCF_2H , Y_1 represents H or F, o represents an integer of 0 or 1, but when o represents 0, and X represents CF_3 , Y_1 represents H.

FIG. 1

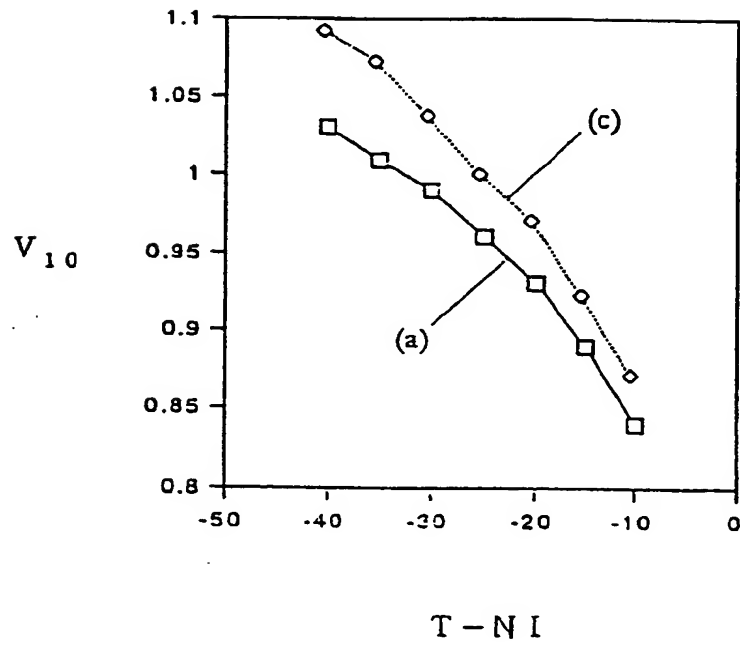
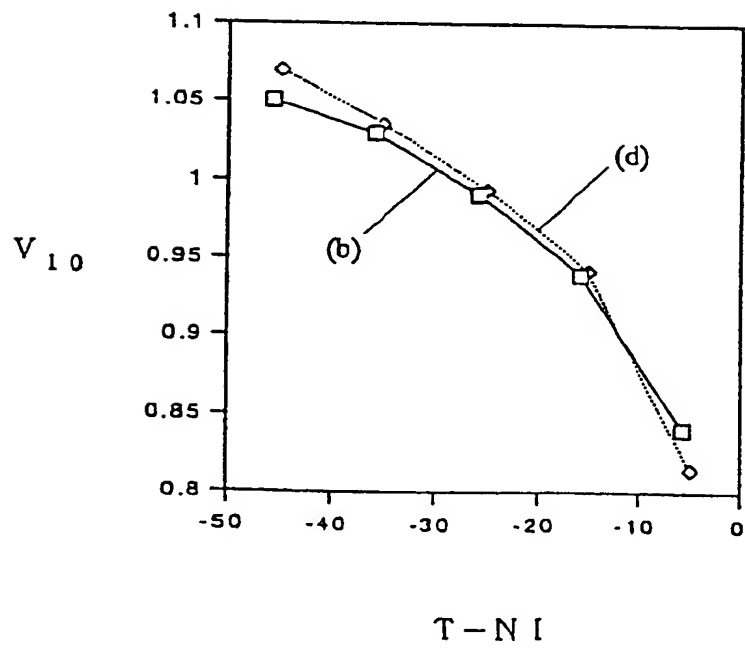
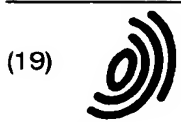


FIG. 2





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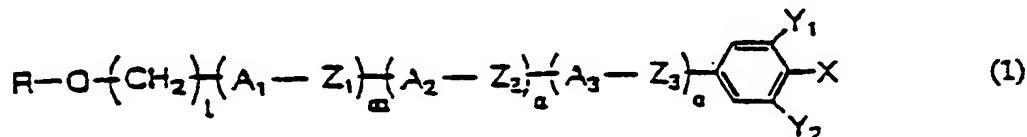
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(57) A novel liquid crystalline compound having a large dielectric anisotropy value, a small change in the threshold voltage depending upon temperatures, and an improved solubility in other liquid crystal materials at low temperatures, and a liquid crystal composition containing the compound are provided,
which liquid crystalline compound is expressed by the formula (1)



wherein R is H or an alkyl group of 1 to 10C; l is an integer of 1 to 9; m , n and o are each independently 0 or 1; A_1 , A_2 and A_3 are each independently trans-1,4-cyclohexylene group, 1,4-phenylene group, one or more H atoms on the ring of which group may be replaced by F, or the like group; Z_1 , Z_2 and Z_3 are each independently -COO-, etc., but at least one member of them is -COO-; X is CF₃, etc.; and Y_1 and Y_2 are each independently H or F.

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EUROPEAN SEARCH REPORT

Application Number
EP 96 10 2672

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Place of search	Date of completion of the search	Examiner	
THE HAGUE	21 May 1997	Beslier, L	
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Application Number
EP 96 10 2672

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Place of search THE HAGUE		Date of completion of the search 21 May 1997	Examiner Beslier, L
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The present search report has been drawn up for all claims			
Place of search THE HAGUE		Date of completion of the search 21 May 1997	Examiner Beslier, L
CATEGORY OF CITED DOCUMENTS X : particularly relevant if taken alone Y : particularly relevant if combined with another document of the same category A : technological background O : non-written disclosure P : intermediate document		T : theory or principle underlying the invention E : earlier patent document, but published on, or after the filing date D : document cited in the application L : document cited for other reasons ----- & : member of the same patent family, corresponding document	

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EUROPEAN SEARCH REPORT

Application Number
EP 96 10 2672

DOCUMENTS CONSIDERED TO BE RELEVANT			
Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int.Cl.6)
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Place of search THE HAGUE		Date of completion of the search 21 May 1997	Examiner Beslier, L
<p>CATEGORY OF CITED DOCUMENTS</p> <p>X : particularly relevant if taken alone Y : particularly relevant if combined with another document of the same category A : technological background O : non-written disclosure P : intermediate document</p> <p>T : theory or principle underlying the invention E : earlier patent document, but published on, or after the filing date D : document cited in the application L : document cited for other reasons ----- & : member of the same patent family, corresponding document</p>			

EPO FORM 150 01.82 (P4C01)



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CLAIMS INCURRING FEES

The present European patent application comprised at the time of filing more than ten claims.

- ☐ All claims fees have been paid within the prescribed time limit. The present European search report has been drawn up for all claims.
- ☐ Only part of the claims fees have been paid within the prescribed time limit. The present European search report has been drawn up for the first ten claims and for those claims for which claims fees have been paid.
namely claims:
- ☐ No claims fees have been paid within the prescribed time limit. The present European search report has been drawn up for the first ten claims.

LACK OF UNITY OF INVENTION

The Search Division considers that the present European patent application does not comply with the requirement of unity of invention and relates to several inventions or groups of inventions.

namely:

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- ☒ All further search fees have been paid within the fixed time limit. The present European search report has been drawn up for all claims.
- ☐ Only part of the further search fees have been paid within the fixed time limit. The present European search report has been drawn up for those parts of the European patent application which relate to the inventions in respect of which search fees have been paid.
namely claims:
- ☐ None of the further search fees has been paid within the fixed time limit. The present European search report has been drawn up for those parts of the European patent application which relate to the invention first mentioned in the claims.
namely claims:



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EP 96 10 2672 - B -

LACK OF UNITY OF INVENTION

The Search Division considers that the present European patent application does not comply with the requirement of unity of invention and relates to several inventions, or groups of inventions, namely:

1. Claims 1-13 (partly) : Compounds of formula 1 wherein $X=CN$ and $Y1=Y2=F$ and their use in liquid crystal compositions.
2. Claims 1-14 (partly) : Compounds of formula 1 wherein X represents CF_3 , CF_2H or CFH_2 and their use in liquid crystal compositions. Starting materials of formula 1-a wherein X represents CF_3 , CF_2H or CFH_2 .
3. Claims 1-14 (partly) : Compounds of formula 1 wherein X represents OCF_3 or OCF_2H and their use in liquid crystal compositions. Starting materials of formula 1-a wherein X represents OCF_3 or OCF_2H .

